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DRAFT SAMPLING AND ANALYSIS PLAN

**Limited Phase II Targeted Brownfields Assessment
Cannery Redevelopment Area
City of Hayward
Hayward, California**

Prepared By:

Innovative Technical Solutions, Inc. (ITSI)
2855 Mitchell Drive, Suite 111
Walnut Creek, CA 94598

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ITSI Project Manager: Jeff Hess
ITSI QA Manager: Jim Schollard

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1.0 INTRODUCTION

The City of Hayward has established the Hayward Redevelopment Plan, which was amended in 1998 to include the revitalization and redevelopment of some of the oldest industrial areas in Hayward. The Cannery Redevelopment Area is one such area (Figure 1).

The Cannery Redevelopment Area has been geographically divided into three sub-areas; Area 1, Area 2, and Area 3. Portions of the Cannery Redevelopment Area have a long history of industrial use, with some sites dating from at least the early 1890s. Although the more recent industrial activities have centered around cannery operations (Areas 1 and 2) and automotive related services (Area 3), past uses have included battery manufacturing (Area 2), bus manufacturing and assembly (Area 2), furniture refinishing (Area 3), and metal plating operations (Area 3). The groundwater beneath the southern portion of Area 2 was identified as being impacted by chlorinated solvents, including tetrachloroethylene (PCE), trichloroethylene (TCE), and 1,1,1-trichloroethane (1,1,1-TCA).

The proposed scope of work of this Limited Phase II Targeted Brownfield Assessment is to conduct a limited groundwater investigation of an area upgradient of Area 2 only. The groundwater upgradient from the southern portion of Area 2 with impacted groundwater will be sampled to evaluate current concentrations of chlorinated solvents relative to current and potential future land use.

A cone-penetrometer (CPT) rig will be employed to drill and geophysically log one subsurface boring. The data will be used to help define the subsurface stratigraphy and assist in determining the optimal depth for collecting groundwater samples. Subsequently, six to eight offsite CPT borings will be drilled and grab groundwater samples will be collected from the desired depths. The groundwater samples will be analyzed for VOCs by EPA method 8260.

1.1 SITE NAME OR SAMPLING AREA

Area 2, Cannery Redevelopment Area, City of Hayward, California.

1.2 SAMPLING AREA LOCATION

Grab groundwater samples will be collected from borings located to the east to northeast of Area 2 (Figure 1). The borings will be located upgradient along the expected groundwater flow direction from the southern portion of Area 2. The borings will be located in low density residential areas, in public right-of-ways along residential streets

1.3 RESPONSIBLE AGENCY

The U.S. Army Corps of Engineers (USACE) has retained ITSI to conduct this Limited Phase II Targeted Brownfields Assessment (TBA) on behalf of the U.S. Environmental Protection Agency (USEPA), as part of a Brownfields Targeted Assessment Program grant to the City of Hayward.

1.4 PROJECT ORGANIZATION

Title/Responsibility	Name	Phone Number
EPA Project Manager	Mr. Jim Hanson	(415) 744-2237
Project Manager (ITSI)	Mr. Jeffrey Hess	(925) 256-8898 x104
Staff (ITSI)	Mr. Rob Nelson Mr. Mark Sorensen	(925) 256-8898 (925) 256-8898 x117
Quality Assurance Manager (ITSI)	Mr. Jim Schollard	(925) 256-8898 x107
Contract Laboratory (Chromalab)	Ms. Surinder Sidhu	(925) 484-1919

1.5 STATEMENT OF THE SPECIFIC PROBLEM

The primary concern for the proposed work is the occurrence of chlorinated VOCs in the groundwater beneath the southern portion of Area 2 at concentrations above the Risk-Based Screening Levels (RBSLs) established by the San Francisco Bay Region of the California Regional Water Quality Control Board (RWQCB). The compounds of concern are; PCE (up to 35 parts per billion [ppb]), TCE (up to 150 ppb), and 1,1,1-TCA (up to 230 ppb).

The full extent of the chlorinated VOC groundwater contaminant plume beneath the southern portion of Area 2 has not been defined. Two sub-plumes appear to be present: 1) a 500-foot-wide plume containing 1,1,1-TCA and 1,1-DCE that extends across a southern portion of Area 2 in a southwesterly direction and 2) a 1,000-foot-wide plume containing PCE and TCE that has a

similar distribution as the 1,1,1-TCA/1,1-DCE plume, but is offset to the south. Groundwater flow beneath Area 2 is expected to be toward the west to southwest.

2.0 BACKGROUND

Area 2 of the Cannery Redevelopment Area has been the host of industrial and agricultural activities since the 1890s, when the first fruit cannery operations began.

2.1 Area Description

Area 2 occupies 67 acres in an urban industrial area. The site is bordered on the north by West A Street, on the west by Amador Street and the Union Pacific railroad tracks, on the south by Winton Avenue, and on the east by Burbank, Myrtle, and Filbert Street. The current redevelopment plans for Area 2 include medium to high density residential areas, live-work areas, parks, a school and community center, and a small commercial area

Current uses of the site encompass the following;

- Amtrak station near A Street and the Union Pacific railroad tracks, consisting of a small platform and an asphalt parking lot
- Cannery Park located west of Burbank between B and C Streets, a city park with an open grassy area, ball fields, and a public restroom facility
- Vacant parcel located between Cannery Park and the railroad tracks
- United Can facility at 199 C Street, a can manufacturing operation consisting of a large brick structure which was part of the former Hunt Brothers Cannery, various outbuildings, aboveground storage tanks (USTs), and supporting facilities
- Filbert warehouse at 201 C Street, a large warehouse and distribution facility
- GSC Logistics warehouse at 24 Cannery Court, a large concrete tilt-up structure
- Foster Farms Dairy at 21 Cannery Court, a wooden structure with truck docks
- Union Pacific railroad right-of-ways extending northwest-southeast between A Street and Winton Avenue
- Burbank School at 353 B Street, including a main school building, numerous portable classrooms, and playground areas; vacant land along A Street between Burbank Street and Meekland Avenue
- Select Foods Inc. 22700 Amador Street, including a cold food storage facility, a recently-abandoned ice company and a drum company
- Centennial Park, located south of Select Foods between Amador Street and the railroad tracks, consisting of open grassy areas, ball fields, and a parking area

- Residential areas along B Street east of Meekland Avenue, along C Street west of Filbert Street, along Myrtle Street north of Winton Avenue, and along Amador Street opposite Elmwood Lane.

Sample Area Description

The area to be sampled is the residential area to the east and northeast (upgradient) from the southern portion of Area 2. The purpose of collecting the groundwater samples is to evaluate the extent and concentrations of chlorinated VOCs in groundwater adjacent to and upgradient from Area 2.

2.2 Operational History

A detailed description of the operational history for Area 2 is presented in the *Phase I Report, Targeted Brownfields Assessment, Cannery Redevelopment Area, City of Hayward*, dated May 25, 2001. The information presented below for operational histories of facilities located in the southern portion of Area 2 is summarized from the Phase I Report. The facility at 199 C Street was operated by the Hunt Brothers Canning Company as a fruit cannery since the early 1890s. During the peak of the cannery operations, the Hayward plant was the largest fruit and vegetable canning facility in the world. Full production at the site ceased in 1979. Many of the older site buildings were demolished at that time, and other structures in the northern portions of the cannery were demolished by approximately 1991; the large warehouse and associated structures are still present.

From 1983 to 1990, the 24 Cannery Court building was occupied by several furniture companies and housed a furniture distribution warehouse, with a small furniture refinishing operation in the northeastern portion of the building. TCA was used in the 1980s for furniture refinishing in the northeastern corner of the 24 Cannery Court facility. Since 1990, various trucking and distribution companies have occupied the facility. Foster Farms Dairy has operated at this site since 1992.

2.3 PREVIOUS INVESTIGATIONS/REGULATORY INVOLVEMENT

A detailed description of the previous investigations is presented in the *Phase I Report, Targeted Brownfields Assessment, Cannery Redevelopment Area, City of Hayward*, dated May 25, 2001.

The information presented below regarding previous groundwater investigations in the southern portion of Area 2 is summarized from the Phase I Report. Groundwater sampling has not been conducted in the residential areas upgradient of the southern portion of Area 2 proposed for sampling under this Limited Phase II TBA. However, soil and groundwater sampling has been conducted in the area of the VOC groundwater contaminant plume within the southern portion of Area 2. Several phases of investigation have been conducted in recent years where the plume passes beneath the southern portion of Area 2, as discussed below.

United Can (199 C Street)

A 300-gallon UST was removed from this site in 1986. UST #4 held gasoline for a fire pump and was located south of the main facility building near the water tower and large aboveground water tanks. In January 1997, the RWQCB issued a closure letter for the 300-gallon gasoline UST at the site.

A subsurface investigation was conducted at a sump in the Lift Truck Shop, located at the southeast end of the main facility building. The sump was removed in May of 1996. Soil samples taken from the excavation contained low concentrations of PCE only. According to the Hayward Fire Department (HFD), the Lift Truck Shop Sump has not received closure from regulatory agencies.

21-24 Cannery Court

Three USTs were present at the 21 Cannery Court site, including two 10,000-gallon diesel USTs and one 1,000-gallon gasoline UST. The USTs reportedly replaced previous USTs located at the site. A groundwater monitoring well had been installed adjacent to the USTs in 1986 as part of a subsurface investigation of a petroleum release. In May 1990, the three USTs, a fuel dispensing island, and associated product lines were removed from the 21 Cannery Court site. Petroleum hydrocarbons were detected in soil and groundwater beneath the site. In April 1997, the RWQCB issued a closure letter for the former USTs at the Cannery Court site.

During the UST investigation at Cannery Court, chlorinated VOCs were identified in the groundwater across the site. Investigations of potential on-site sources revealed no detections of

VOCs in the soil. The highest concentrations of VOCs were detected in groundwater samples from the upgradient wells. Groundwater was determined to flow in a south-southwesterly direction, with groundwater occurring between approximately 44 to 52 feet bgs.

The investigations concluded that the distribution of VOCs across the site indicated an upgradient off-site source, and there was no evidence the Cannery Court site was contributing to the plume. The RWQCB reviewed potential upgradient sources and did not identify any upgradient or cross-gradient sites with similar VOC concentrations in the groundwater. The RWQCB concurred with the conclusion regarding an off-site source. All monitoring wells at the site were abandoned in May 1996 and August 1997.

2.4 GEOLOGICAL INFORMATION

Area 2 is located in the greater San Francisco Bay Area of the Pacific Coastal Range geomorphic province, which is characterized by a mixture of marine and nonmarine sedimentary rocks that are overlain by alluvial materials (RWQCB, 1999). The site is located within the San Lorenzo alluvial cone hydrogeologic unit of the East Bay Plain (E&K, 1993). The region is bounded by the San Leandro alluvial cone to the north, the foothills of the Diablo Range to the east, the Niles cone to the south, and San Francisco Bay to the west. The East Bay Plain includes an alluvial area near the foothills of the Hayward Hills and a marshland area adjacent to the San Francisco Bay (Golder, 1996).

The San Lorenzo alluvial cone consists of a series of coalescing alluvial fan deposits derived from the drainage basin of San Lorenzo Creek, which drains the western slope of the Diablo Range. The deposits in the area consist of a mixture of poorly consolidated to unconsolidated clay, silt, sand and gravel. Shallow, unconfined and discontinuous perched aquifers and clay beds extend to a depth of approximately 50 feet bgs. Water levels in the shallow aquifers may vary seasonally. The shallowest confined aquifer in the vicinity of Area 2 is reportedly the Newark Aquifer, which is located at a depth of approximately 50 to 130 feet bgs and is separated from the shallow unconfined aquifers by a clay aquitard (Golder, 1996). Below the Newark Aquifer, the Centerville and Fremont aquifers are found to depths up to 400 feet bgs; other aquifers below 400 feet comprise the lower aquifer zone.

Groundwater in the San Lorenzo alluvial cone is used primarily for industrial water supply and irrigation (RWQCB, 1999). The shallow aquifers are generally used for irrigation only. The City of Hayward acquires its drinking water from the City and County of San Francisco Water Department's Hetch Hetchy Aqueduct. Before the 1950's, groundwater was the main source for the City's water supply. The City's emergency water supply system uses groundwater, and a water-supply well is located at the Alameda County complex approximately 0.4 miles west of Area 2. The total depth of this well is 560 feet, with the screened interval in the lower aquifer zone.

The groundwater gradient established during site investigations conducted in and surrounding Area 2 is generally west to southwest, which is consistent with the regional groundwater flow direction (RWQCB, 1999). Groundwater in the northeastern section of Area 2 was reported to flow in a west to southwesterly direction, and depths to groundwater ranged from approximately 36 to 47 feet bgs (IT, 1996). In the southeastern portion of Area 2, groundwater was determined to flow in a south-southwesterly direction, with groundwater occurring between approximately 44 to 52 feet bgs (E&K, 1993).

2.5 ENVIRONMENTAL AND/OR HUMAN IMPACT

The presence of the VOCs in the groundwater beneath the southern portion of Area 2 could adversely impact unrestricted use of this area. Vapor-phase VOCs present in soil gas in equilibrium with a groundwater plume can migrate upward through the soil and adversely affect indoor air quality in enclosed structures located above the plume. Groundwater investigations concluded that the VOCs originated from an upgradient off-site source. There is currently no ongoing investigation of the VOC plume underlying the southern portion of Area 2.

A review of prior groundwater monitoring reports indicates a potential for two different plumes, a PCE/TCE plume centered along Myrtle Street and a 1,1,1-TCA/1,1-DCE plume centered a little to the north along Meek Avenue. The extent of this plume is undefined. The distribution of the PCE/TCE plume suggests a possible distant source, but the TCA/1,1-DCE plume is narrower and could represent a more nearby source. It should be noted that relatively high levels of PCE and TCE have recently been identified at the American Stores site, located upgradient and

approximately 3,200 feet northeast of Area 2. However, 1,1,1-TCA and 1,1-DCE have not been detected at the American Stores site.

VOCs in the groundwater beneath the southern portion of Area 2 exceed applicable RBSLs as established by the RWQCB. However, based on discussions with the RWQCB, the RBSLs for chlorinated VOCs are based on drinking water, as these values are considered the most conservative level of risk. The RBSLs based on drinking water impacts could be addressed through restricting groundwater use in the existing residential areas and within Area 2. However, the most-recent VOC results are from 1994. Since no contaminant source has been identified, the potential for an upgradient source continuing to contribute to the plume(s) exists. Discussions with the RWQCB indicate further definition of upgradient VOC concentrations would be required to fully evaluate the potential future risks from the VOCs in the groundwater and the associated impacts to residential development of Area 2.

3.0 PROJECT DATA QUALITY OBJECTIVES

3.1 PROJECT TASK AND PROBLEM DEFINITION

The purpose of this proposed Limited Phase II TBA investigation is to define the presence and concentrations of chlorinated VOCs in groundwater located upgradient (generally northeast) of the southern portion of Area 2. The collection of groundwater samples will help to determine both the concentrations of contaminants beneath the southern part of Area 2, as well as whether higher contaminant concentrations from off-site sources are approaching Area 2 from the upgradient direction.

Grab groundwater samples from six to eight soil boring locations will be collected and analyzed for full-scan VOCs by EPA method 8260. The groundwater sampling results will be used to perform a Tier 2 RBSL analysis per RWQCB guidelines of potential impacts to human health posed by the presence of chlorinated VOCs in groundwater. The concern is that VOCs originating from a groundwater contaminant plume can volatilize from the plume into soil gas, migrate upward through the unsaturated soil, diffuse into structures such as houses, and create an inhalation threat.

3.2 DATA QUALITY OBJECTIVES (DQOs)

The following is a brief description of the seven-step Data Quality Objectives (DQO) process used in developing the scope of work outlined in the SAP:

- | | |
|---|---|
| Step 1: Identification of the problem | Evaluate the presence and concentrations of groundwater contaminants adjacent to and upgradient from the site (the southern portion of Area 2). |
| Step 2: Identification of decisions | Are groundwater contaminants present at concentrations which could affect the future use of the property? |
| Step 3: Identify the inputs to the decision | The inputs will be the analytical and field data collected during the monitoring and sampling programs. |
| Step 4: Definition of study boundaries | The limits of the study area are the area upgradient of the southern portion of Area 2. |

Step 5: Development of decision rules	If hazardous materials are identified in the groundwater at concentrations above the RBSLs or MCLs unrestricted use of the southern portion of Area 2 may be impacted.
Step 6: Specification of limits on decision errors	Accuracy and precision of laboratory results will be within EPA-defined limits. Detection limits not to exceed applicable RBSLs and MCLs.
Step 7: Optimization of investigative design for obtaining data	Advance soil borings with a cone-penetrometer rig at locations designated in workplan. Perform lithologic logging at the first boring location using CPT to determine the subsurface stratigraphy as well as optimal depth for groundwater sample collection. At subsequent locations, collect groundwater samples for VOC analysis from the upper portion of the saturated zone.

Specific criteria for accuracy, precision, and completeness will be utilized during this project, and specific limits are discussed in their respective sections within this SAP.

The applicable RBSLs for the chlorinated VOCs are listed in the following table. Based on discussions with the RWQCB, the groundwater RBSLs for chlorinated VOCs are based on drinking water MCLs, as these values are considered the most conservative level of risk. However, a primary concern for the residential development of the site is the accumulation of VOCs in indoor air, as a result of groundwater contamination. The table below also shows the indoor air component of the RBSLs for the chlorinated VOCs found in the groundwater. These indoor-air RBSLs represent subsurface values for the sites where the contamination is located at a depth greater than 10 feet bgs, and where fine-grained, less permeable soils are present.

Chemical of Concern	Site Data	RBSL-groundwater	RBSL – Indoor Air Impacts	RBSL-Tier 2
PCE	35 ppb in groundwater	5.0 ppb	15,000 ppb in groundwater	To be determined
TCE	150 ppb in groundwater	5.0 ppb	57,000 ppb in groundwater	To be determined
TCA	230 ppb in groundwater	62 ppb	1,300,000 ppb in groundwater	To be determined
1,1-DCE	66 ppb in groundwater	6.0 ppb	1,000 ppb in groundwater	To be determined

3.3 DATA QUALITY INDICATORS (DQIS)

Data quality indicators (accuracy, precision, completeness, representativeness, comparability, and method detection limits) refer to quality control criteria established for various aspects of data gathering, sampling, or analysis activity. The QC criteria established for each indicator is discussed below.

Accuracy

Accuracy is the degree of agreement of a measurement with a known or true value. To determine accuracy, a laboratory or field value is compared to a known or true concentration. Accuracy is determined by such QC indicators as: matrix spikes, surrogate spikes, laboratory control samples (blind spikes) and performance samples. For Method SW8260B, accuracy will be evaluated through the analysis of MS/MSD samples, LCS, and by spiking all samples with surrogate compounds where applicable. Only investigative samples from delivery order projects or sites will be used for Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses.

For every 20 samples submitted to the contract laboratory, one sample will be provided in sufficient quantity to allow sample analysis, MS/MSD analysis, and re-extraction/re-analysis of the MS/MSD. ITSI will select or identify samples for MS/MSD analysis that are representative of site conditions and analytes of concern. Field-generated blanks will not be used for MS/MSD analyses.

The MS/MSD samples will be spiked with the method target compounds indicated in Table 3-1; a third aliquot of the sample will be analyzed unspiked. Accuracy will be measured in terms of

the percent recovery of the spiked compounds. If MS/MSD analyses do not meet the recovery criteria specified in Table 3-1, both the MS and MSD samples will be re-extracted and re-analyzed. Both the MS and MSD recoveries must be within acceptance criteria for this QC requirement to be satisfied (i.e., averaging of the MS/MSD recoveries is not allowed).

Failure of MS/MSD analyses to meet QC criteria shall result in the initiation of a review of the corresponding analytical batch to determine if the failing spike result is an isolated event or indicative of an analytical batch failure. Re-extraction and/or re-analysis of the entire analytical batch may be required if data indicate that the analytical system is not functioning properly. Samples will be spiked with the surrogate compounds indicated in Table 3-1. If surrogate compound analyses do not meet the recovery criteria specified in Table 3-1, the sample will be re-extracted and reanalyzed.

LCS analyses are spikes on a blank matrix to assess accuracy independent of matrix effects. Blank matrices are deionized water for water samples. The LCS will be spiked with the method target compounds indicated in Table 3-1. If LCS analyses do not meet the recovery criteria specified in Table 3-1, the LCS sample will be re-analyzed to determine if the failure is due to a transient instrumental condition. If the second analysis does not meet the recovery criteria, the LCS and the entire analytical batch will be re-extracted and re-analyzed within the holding time.

Precision

Precision is the degree of mutual agreement between or among independent measurements of a similar property (usually reported as a standard deviation [SD] or relative percent difference [RPD]). This indicator relates to the analysis of duplicate laboratory or field samples. Typically field precision is assessed by co-located samples, field duplicates, or field splits and laboratory precision is assessed using laboratory duplicates, matrix spike duplicates, or laboratory control sample duplicates. For Method SW8260B, precision will be evaluated through the analysis of field duplicate samples and MS/MSD samples. Field duplicate samples will be collected at a frequency of one duplicate per 10 samples; the identity of field duplicate samples will not be provided to the laboratory, and samples will not be re-analyzed when field duplicate criteria are not met.

Precision will be measured in terms of the RPD of the field duplicate samples and the RPD of the MS/MSD recoveries. RPD criteria are specified in Table 3-2. If MS/MSD analyses do not meet the RPD criteria, the MS/MSD samples will be re-extracted and re-analyzed. Field duplicate results will not be used to quantitatively assess completeness.

Completeness

Completeness is expressed as a percentage of valid usable data actually obtained compared to the amount that was expected. Due to a variety of circumstances, sometimes either not all samples scheduled to be collected can be collected, or the data from samples cannot be used (for example, samples lost, bottles broken, instrument failures, laboratory mistakes, etc.). Qualitative completeness will be assessed by reviewing the tasks that contribute to the sampling event such as chain of custody procedures, adherence to the work plan, and adherence to the sampling and analysis plan. Quantitative completeness is defined as the ratio of the number of the acceptable sample results obtained to the total number of sample results collected. The QC parameters to be evaluated in determining quantitative completeness are: holding times, initial calibrations, continuing calibrations, surrogate recoveries, LCS recoveries, MS/MSD recoveries and RPDs, and laboratory duplicate RPDs. For Method SW8260B, the minimum percent completeness goal is 90 percent. However, evidence of legitimate matrix effects that is accepted by the EPA will not be counted against completeness.

Representativeness

Field Procedures

Field procedures representativeness refers to the collection of samples that allow accurate conclusions to be made regarding the composition of the sample media at the entire site. Representativeness will be qualitatively assessed by evaluating whether the procedures described in this SAP were followed. Specifically, the site-sampling layout, including sampling locations, frequency of sampling, and timing of sampling activities, will be reviewed. In addition, sample collection, packaging, and preservation procedures will be reviewed. In particular, sample preservation will be monitored closely for conformance to requirements in this SAP.

Laboratory Procedures

To obtain the sample for analysis by Method SW8260B, the contract laboratory will transfer a sample aliquot from a VOA vial containing no headspace. In addition, laboratory procedures will be reviewed to verify that standard operating procedures were followed and method requirements were met during the analysis of project samples. Specifically, laboratory sample storage, holding times, sub sampling procedures, method blanks, and evidence of matrix interference will be assessed.

Comparability

Comparability expresses the confidence with which one data set can be compared to another. The use of methods from EPA or "Standard Methods" or from some other recognized sources allows the data to be compared facilitating evaluation of trends or changes in a site, a river, groundwater, etc. Comparability also refers to the reporting of data in comparable units so direct comparisons are simplified. To ensure data comparability, the contract laboratory will follow standard test methods (Method SW8260B) and means of sample preservation; standard units, detection limits, calculation procedures, and reporting formats; and standard measures of accuracy and precision.

Method Detection Limits

The laboratory must conduct an MDL study for all Method SW8260B target analytes listed in Table 3-3 using the protocol established in 40 CFR Part 136, Appendix B. If multiple instruments are used to analyze project samples, all instruments must be included in the MDL study and the reported MDL should be representative of the least-sensitive instrument or the specific instrument used for analysis, depending on the requirements of the project governing documents. MDL studies must be updated annually. The laboratory will be required to submit the most current MDL studies to ITSI as they are completed to demonstrate its capability to achieve the maximum allowable PQLs listed in Table 3-3.

3.4 DATA REVIEW AND VALIDATION

Given that the sample set is small (<10 samples) and that samples will be collected within two field days, 100 percent of the data will be validated by the ITSI project chemist. A traditional full validation (EPA Tier 3) will be performed. In each data package, the contract laboratory will provide the following information for validation:

Case Narrative, to include:

- A cross-reference table of ITSI field sample numbers and laboratory identification numbers, including the sample collection and laboratory receipt dates.
- A summary of the analytical method(s) performed.
- A summary of sample receipt problems, changes in analytical requirements, and analytical anomalies.
- A discussion of all QC issues associated with the analytical batch, including: QC samples, surrogates, and internal standards with recoveries outside of control limits; holding time violations; sample dilutions; and sample re-analyses due to QC problems. All related laboratory corrective actions must be addressed in the case narrative.
- A glossary of all symbols, terms, and data flags used in the laboratory report.

Analytical Results, to include:

- Preparation and analysis method numbers
- Sample matrix, ITSI identification number, and the laboratory identification number
- Dates for sample collection, receipt, preparation, and analysis
- Instrument identification and dilution factor
- List of analytes with quantitative results, units, MDL, and PQL (result summary report)
- Quantitation report
- Chromatograms
- Dual mass spectra for all positive results
- Surrogate recoveries and control limits
- Summary of internal standard recoveries and control limits
- Explanation of all qualified results
- Results for sample re-analyses (including dilutions) will be reported separately. For dilutions, both results may be reported on hard copy.

QC Results, to include:

- A cross-reference table of laboratory batch identification for all QC samples and associated field samples with batch preparation and analysis dates

Method blank results for all analytical batches, including:

- Quantitative results for all target analytes

- Chromatograms
- Mass spectra for all positive results
- Internal standard recoveries, retention times, and control limits
- MDLs
- PQLs.

Spiking results for all analytical batches for LCS, LCSD, MS, and MSD as applicable, including:

- Quantitative results, spiking levels and percent recoveries for all spiked analytes
- Control limits for accuracy and precision
- Identification of MS parent samples
- Chromatograms
- Mass spectra for all positive results
- Internal standard recoveries, retention times, and control limits
- Flagged outliers.

Surrogate recovery summary, including:

- Spiking levels for all surrogates
- Percent recoveries
- Acceptance criteria
- Flagged outliers.
- Copies of MDL studies associated with the results submitted in the data package.
- Retention time window summary for all target analytes, surrogates, and internal standards.

COC and Cooler Receipt Forms

- Signed original copies of the COC forms showing date and time of sample receipt.
- Cooler receipt form.

Initial Calibration, to include:

- Instrument identification
- Standard preparation logs
- Date and time of calibration
- Calibration standard concentrations

- Calibration model including equation and constants used to calculate results
- Quantitation reports
- Chromatograms
- Response factors and percent relative standard deviation (RSD) summaries or least-squares fit of data with calculated regression concentrations
- Acceptance criteria
- Flagged outliers.

Continuing Calibration, to include:

- Instrument identification
- Response factor and percent difference (D) summaries
- Calibration standard concentrations
- Quantitation report
- Chromatograms
- Standard preparation logs
- Date and time of calibration
- Acceptance criteria
- Flagged outliers.

Internal Standard Summary, to include:

- Standard preparation logs
- Spiking levels for all internal standards
- Retention times and areas for all internal standards in field and QC samples
- Results for reference calibration standard
- Acceptance criteria
- Flagged outliers
- Raw data, calculations, and data determined to support reported MDLs.

Tune Summary, to include:

- Instrument identification
- Instrument tune data (ion abundance table) with acceptance criteria and a summary of the associated standards, laboratory QC, and sample numbers
- Tune spectra and mass listings
- Date and time of analysis

- Acceptance criteria
- Flagged outliers
- Chronological listing of associated field and QC samples with analysis times.

Log Sheets, to include:

- Extraction bench sheets
- Injection logs.

Procedures to Validate Data Integrity

Data integrity is dependent on the ability of ITSI and the analytical laboratory to effectively implement QA/QC procedures identified in this SAP. Data integrity is validated throughout the project by evaluating the degree of adherence to SAP requirements. Laboratory review of analytical data will be in conformance with EPA Guidance Document SW-846 (EPA, 1994) and laboratory standard operating procedures.

The ITSI project chemist will validate the data for the parameters indicated below:

- Sample Integrity
- Technical Holding Times
- Instrument Tunes
- Initial and Continuing Calibrations
- Analyte Identification
- Analyte Quantitation
- Method Blanks
- LCS/LCSD Accuracy and Precision
- MS/MSD Accuracy and Precision
- Surrogate Recoveries
- Field Duplicate Precision

One hundred percent of laboratory-generated data will be subjected to this internal data review. Matrix interferences manifest themselves as reproducible failure occurrences to meet project QC goals for chemical data. If matrix interferences become apparent during sample analysis, method modifications such as additional cleanup steps, sample volume changes, and analytical procedure

revisions will be attempted and documented. If method modifications do not remedy the problem, alternative procedures will be proposed. The laboratory will assign qualifiers to the data to indicate potential impacts to data use.

The laboratory will be responsible for assuring the following:

- Sample chain of custody documentation is complete and correct
- Sample preparation information is complete and correct
- Sample integrity has been maintained
- Instrument performance criteria have been met
- Calibration criteria have been met
- Holding times, sample preservation, and sample storage criteria have been met
- Analyte identification and quantification are correct
- QC samples and method blanks are within control limits
- Documentation (including the case narrative) is complete and correct
- Reports have been prepared and delivered in accordance with contract requirements.

In the event that the above items are identified as deficient, corrective action will be initiated. Analytical laboratory corrective action items are identified in Table 3-4 of this SAP.

Outliers

Identification and treatment of outlying data may occur at any phase of the data collection process. Once identified, outlying data is characterized with respect to the cause, nature, and degree of the QA/QC exceedances.

Corrective action measures specified in Table 3-4 of the SAP will be taken to resolve problems and restore proper function to the analytical system when data indicate that the analytical system is not performing adequately. These measures may be necessary when the following occurs:

- QC data are not within the control limits for precision and accuracy
- Blanks contain contaminants above the acceptable levels

- Calibration data or instrument performance parameters are not within acceptance criteria (Table 3-5).
- Undesirable trends are observed in the QC data or calibration data
- There are unusual changes in instrument sensitivity or performance

Initiation of corrective action resulting from the evaluation of QC results will be conducted by the laboratory QA manager in consultation with the ITSI Project Chemist. Corrective actions are identified in Table 3-4 and may include, but are not limited to the following:

- Re-analysis of the samples
- Documentation of interferences or matrix effects that result in poor analytical performance
- Evaluation and initiation of sample extract clean-up procedures
- Evaluating and changing sampling or analytical procedures
- Resampling and re-analysis, if the completeness or usability of the data set does not meet the criteria for acceptability.

Data that do not meet data quality objectives will be qualified according to the data validation procedures presented in Table 3-6.

3.5 DATA MANAGEMENT

Data management includes procedures for generating, tracking, reporting, and storing data acquired during contract activities. Data are generated during the field program and the analytical laboratory testing process. Due to the small data set, field data will be recorded in bound notebooks and manually transferred to an electronic spreadsheet. Laboratory data will be provided as hardcopy reports, with the data manually transferred to an electronic spreadsheet.

Data can be updated or modified, and publication-quality reports can be created using project-specific spreadsheets or databases, as appropriate. The data can be automatically exported in a variety of formats from the spreadsheets or databases for use in AutoCAD, geographic information systems (GIS), or other applications, as appropriate. Data validation may be facilitated through an automated QA/QC reporting system prior to manual review of reports, as appropriate.

Data entered either manually or electronically into spreadsheets and/or database are reviewed for accuracy. If discrepancies are discovered during the review process, the cause is identified and corrective action is implemented by the project chemist or project manager. At the next appropriate opportunity, the project chemist or project manager confirms that the discrepancy or error has been resolved. Data are not loaded into the spreadsheets or database until they have passed a stringent set of manual and/or automated error detection procedures, including checking:

- Analyses requested on the chain of custody forms with the analyses reported by the laboratory
- Syntax to assess whether the correct types of data are in the appropriate fields
- Whether the data provided by the laboratory contain the required entries
- Preparation and test method codes against a project-specific list
- Laboratory QC sample codes against a project-specific list
- Database output to make sure that reports accurately represent analytical results as reported by the analytical laboratory.

Access to the spreadsheets and/or database is restricted to authorized personnel responsible for data management. Typically only the project chemist, project manager or designated alternates are allowed to update or modify files in the spreadsheets and/or database.

Data Archiving

An electronic backup copy of the chemical data spreadsheets and/or databases is made weekly and stored in a fire-resistant location. Hardcopy data reports are stored in the ITSI office managing the contract, according to project, site, and laboratory batch identifiers, and are kept for a minimum of 5 years.

The contract laboratory shall preserve all information regarding sample analyses (calibration records, etc.) such that the analytical process can be reconstructed in the future. This will include the ability to reconstruct chromatography for organic methods.

Other data stored in spreadsheets and/or databases (e.g., chain of custody information, water-level and survey data, and boring log files) are archived using both hard copy and electronic media. Periodic backups of these electronic files are stored on removable media (i.e., diskette, CD-R, DAT) and are kept for a minimum of 5 years in the ITSI office managing the contract. Raw data and analysis records associated with samples collected for the contract will also be stored by the contract laboratory for a minimum of 5 years.

3.6 ASSESSMENT OVERSIGHT

The overall QA program relies on the unambiguous assignment of responsibilities. The implementing corporate and/or project-specific QA/QC requirements are as follows:

- Project Manager: Responsible for implementation of project activities and three-phase quality control for each definable feature of work.
- Task manager: Reports to the project manager(s) and is responsible for implementing and overseeing project QA/QC activities. Directly monitors the work of field subcontractors and activities such as, soil boring and well installation.
- Project chemist: Reports to the project manager and is responsible for analytical laboratory subcontractor performance and implementation of three-phase quality control of the analytical activities.
- Project staff: Report to task manager and are responsible for implementing project activities according to plans, instruction, and requirements.

ITSI utilizes appropriate project management tools for developing project schedule, timeline, and deliverable requirements for each project. The project manager must see to it that appropriate project schedules are established, communicated to the USACE, and met in a timely fashion. Prior to submittal to the USACE, all documents and work products prepared by ITSI will undergo multiple levels of review that include but are not limited to peer review, technical editing review, QC review, and project manager review. ITSI has a dual signature policy that requires, at a minimum, signatures of the task manager and project manager on the document. In addition, formal reports will be signed by the program manager.

Three-Phase Quality Control

ITSI utilizes the three-phase quality control approach, including preparatory, initial, and follow-up phases, to monitor adherence to project requirements. Field or laboratory deficiencies that

have been identified during project performance, audits, and reviews are addressed by the project manager, project chemist, or other individuals as appropriate. Identification of deficiencies and corrective actions are documented by written communications such as memoranda, telephone conversation records, electronic mail, or letters, addressed or forwarded to the USACE technical manager assigned to the project. As necessary, such documentation will become part of the project record or report.

Three-phase quality control for data acquisition includes the development of project planning documents, communication of project plans with the analytical laboratory and field staff, collection of samples and field data during field operations, analysis and reporting of analytical data by the laboratory, and review and validation of the data after reporting. Three-phase quality control will be implemented for data acquisition activities for each definable feature of work, as listed below. Personnel responsible for different aspects of each phase of the quality control operation are indicated after the description of the quality control activity.

Phase I: Preparatory Phase QC

Preparatory phase QC will be accomplished before work begins on the project and will be to:

- Notify the USACE Technical Manager or Contracting Officer at least 72 hours prior to commencement of preparatory phase QC activities
- Review contract and project specific planning documents including the SAP (Project Manager)
- Review and discuss with subcontractor laboratory the relevant sections of the CDQMP or the SAP. Prepare meeting notes including documentation that corrective action (if necessary) has been implemented. Send copy of meeting notes, including signoff by all participants, to the USACE Contracting Officer (Project Chemist, Subcontract Laboratory)
- Review of applicable specifications from the CDQMP, Statement of Work, SAP (Project Chemist, Project Manager, Task Manager, Project Staff)
- Check field analytical equipment to confirm that the equipment is in good working order and that necessary reference and calibration supplies are available (Project Chemist and Task Manager)
- Examine the work site or area to check that any required preliminary work has been completed in accordance with the SAP (Task Manager)
- Review the appropriate sections of the Site-Specific Health and Safety Plan to make sure that appropriate health and safety procedures will be followed (Task

- Manager, Project Staff, and Subcontractors, as necessary)
- Conduct a planning meeting among field team members regarding procedures for performing the planned activities (Task Manager, Project Staff, and Subcontractors, as necessary)
- Document in the Daily Activity Report (Section 4.3.1) that above quality control items have been addressed during the work (Task Manager or Designated Representative).

Phase II: Initial Phase QC

Initial phase QC will be accomplished at the beginning of each definable feature of the project and will:

- Review the preliminary work to make sure it has been accomplished in compliance with the Statement of Work and the SAP as applicable (Project Manager, Task Manager, Project Chemist, as appropriate)
- Perform audits of field procedures and field analytical techniques as specified in the SAP (Project Chemist, Task Manager)
- Verify that any required audits and inspections have been performed and that corrective actions have been implemented (Task Manager)
- Establish levels of performance and verify compliance with minimum acceptable performance standards as specified in the SAP (Task Manager, Project Chemist)
- Resolve any differences between levels of performance and minimum acceptable performance standards (Project Manager, Task Manager, Project Chemist, as appropriate)
- Review of the appropriate section of the Site-Specific Health and Safety Plan to make sure that correct procedures are followed and to assess whether upgrading of the safety plan will be necessary, based on changes to the initial scope of work (Health and Safety Officer, Task Manager, Project Staff, as appropriate)
- Notify the USACE at least 48 hours before the initial phase of the project begins (Task Manager)
- Repeat steps for the initial phase when specified quality standards are not being met when new staff are added to the project or when modifications to the Statement of Work or SAP are implemented that impact existing field procedures or quality control standards (Project Staff).

Phase III: Follow-Up Phase

The follow-up phase includes periodic checks that will be performed to assess the degree of compliance with contract requirements and will continue until each particular feature of the project is complete. The checks will become part of the project quality control documentation

and deficiencies will be reported as part of the Daily Quality Control Reports to the USACE. Follow-up checks will be conducted. Corrective action will be implemented prior to the start of additional phases of the project that may be affected by the noted deficiencies. Under direction of the Project Manager, the entire project team may be involved with the follow-up phase quality control activities. Additional preparation and initial phase QC activities will be implemented on request of the USACE and when project QA/QC requirements are not met.

Assessments and Response Actions

Assessment and response actions will be conducted by ITSI throughout contract activities. Assessment and response actions are integrated with the three-phase quality control operations described in Sections 3.1 of the QAPP and Section 6.0 of the FSP. Members of ITSI's project team are collectively responsible for assessment activities. All project personnel have authority to implement corrective action or stop work depending on the situation. Examples of project assessment activities are listed below.

- Peer review
- Quality control review
- Checks to see that project personnel have read appropriate planning documents and are following documented procedures
- Checks to see that clearance activities and preliminary work have been satisfactorily completed
- Comparison of data generated with project goals and objectives
- Data review and validation.

Reports To Management

Reports to management will be prepared throughout each project, as appropriate, and may include the following:

<u>Report Type</u>	<u>Schedule</u>	<u>Responsible Person</u>	<u>Recipient of Report</u>
Daily Activity Reports	Throughout project	Task Manager	Project Manager, USACE Contracting Officer
Non-Routine Occurrences Reports	Throughout project	Project Chemist, Project Manager	Project Manager, USACE Contracting Officer
Quality Control Summary Report	Draft report preparation	Project Chemist	Task Manager, Project Manager, USACE Contracting Officer

Telephone
conversation records

Throughout
project

Project staff as
appropriate

Project Manager, Task
Manager

ITSI Daily Activity Reports

For each day of the project in which field activity occurs, ITSI will prepare field Daily Quality Control Reports (DQCRs) from the field sampling notes and observations and audit reports. DQCRs will be transmitted to the USACE on a weekly basis. These reports will contain the following:

- Location of work
- Weather conditions
- Description of work performed
- Problems encountered and associated corrective action
- Any instructions received from USACE personnel for retesting
- Description of field tests performed, including the individuals performing the tests, test results, and calibration procedures
- Results of audits or inspections, including problems identified and corrective actions taken
- General comments

Laboratory Daily Activity Reports

- The laboratory will provide the Task Manager or Project Chemist with DQCRs that will be limited to reporting out-of-control analytical events. These will contain the following:
 - Holding time violations
 - Chain of custody discrepancies
 - Sample storage and preservation errors
 - Out-of-control QA/QC sample results
 - Corrective actions taken as a result of the above problems.

Field and laboratory DQCRs will be assembled and provided to the USACE Project Manager each week; weekly DQCRs will be sent to the USACE on the Tuesday following the week the fieldwork was performed.

Quality Control Summary Report

ITSI will prepare a Quality Control Summary Report (QCSR) at the end of the project. The QCSR will be submitted to the USACE as part of the site or activity summary report or other report deliverable as requested by the USACE in the scope of work for the project. The QCSR will contain the following information:

- Project scope
- Project description
- Sampling procedures
- Analytical procedures
- Data quality assessment, which will present a summary of the data validation and data quality issues for all laboratory test methods. This summary will include a quantitative assessment of analytical laboratory completeness and contract completeness, and a summary of all analytical laboratory and field QA/QC results.
- Conclusions and recommendations, which will summarize the recommended changes to sampling and analytical laboratory procedures to better assess site conditions in future contract work.

In addition, the QCSR will contain summaries of QA/QC and data validation results using ITSI data validation worksheets.

Non-Routine Occurrences Reports

ITSI will provide the USACE Contracting Officer or Project Manager with written reports of significant non-routine occurrence events within 48 hours of notification or knowledge that a non-routine event occurred. Significant events are occurrences that impact the cost, schedule, or quality of the work or the quality of the environmental analytical data. The report will identify the problem, corrective action taken, and any verbal or written instructions received from the USACE Contracting Officer.

4.0 SAMPLING RATIONALE

4.1 SOIL SAMPLING

Not applicable.

4.2 SEDIMENT SAMPLING

Not applicable.

4.3 GROUNDWATER SAMPLING

Groundwater samples will be collected from eight temporary borings drilled into the upper part of the saturated zone at eight locations. The samples will be analyzed for full-scan VOCs using EPA method SW8260B. These analytes were chosen because of the need to acquire data for a screening-level evaluation of current groundwater conditions at the site relative to RBSLs and MCLs.

The borings will be drilled using a cone-penetrometer (CPT) rig. The first boring will be logged using standard CPT methods to partially determine the site's stratigraphy, and to determine the optimal depth at which a groundwater sample can be collected in sufficient yield. A second boring placed near the first boring will be used to collect a groundwater sample from the desired depth within the upper part of the saturated zone. A discrete point sampler with a detachable tip will be driven to the desired sample depth. Then grab groundwater samples will be collected by lowering a disposable bailer within the sampler.

Two borings will be drilled near the southern end of Souza Court and along Alice Street, approximately 200 feet south of the intersection of Alice and Claire Streets. The rationale for these borings is to determine whether the small-industrial area bounded approximately by Alice, Claire, Grand, and C Streets may be a contaminant source for the groundwater contaminant plume under the site (the southern part of Area 2 of the Cannery Redevelopment Area).

Two borings will be drilled near the western end of Pamela Court and along Myrtle Street, approximately 350 feet south of the intersection of Myrtle and Lion Streets. The rationale for

these borings is to characterize groundwater VOC concentrations in the contaminant plume upgradient from the site.

Two borings will be drilled approximately 200 feet west of the intersection of Meek and Myrtle Streets, and approximately 250 feet south of the intersection of Meek and Myrtle Streets. The rationale for these borings is to determine 1) current contaminant concentrations in the contaminant plume beneath the site; and 2) whether the contaminant source for the plume is indeed off-site and upgradient from the Cannery Court facility.

4.4 BIOLOGICAL SAMPLING

Not applicable.

4.4.1 Biological Samples for Chemical Analysis

Not applicable.

4.4.2 Biological Sample for Species Identification and Habitat Assessment

Not applicable.

5.0 REQUEST FOR ANALYSES

5.1 ANALYSES NARRATIVE

As enumerated in Table 5-1, groundwater samples will be taken at eight locations. A double-volume groundwater sample will be collected at an appropriate sample location and used as a laboratory QC sample. A field duplicate groundwater sample will also be collected at an appropriate sample location.

As shown in Table 5-1, each groundwater sample (including laboratory QC samples) will be analyzed for volatile organic compounds by Method 8260B.

5.2 ANALYTICAL LABORATORY

The proposed laboratory is Chromalab located in Pleasanton, California. Chromalab is certified by the State of California. A copy of Chromalab's SOP for Method SW8260B is provided in Appendix A.

6.0 FIELD METHODS AND PROCEDURES

6.1 FIELD EQUIPMENT

6.1.1 List of Equipment Needed

- Water-level indicator
- Discrete-point sampler with detachable tip
- Disposable bailers
- Photoionization detector
- Disposable gloves

6.1.2 Calibration of Field Equipment

The only field equipment requiring calibration is the photoionization detector, which is calibrated daily in the field. The instrument is calibrated according to manufacturer's specifications, first using zero (clean) air to calibrate zero, followed by placing the probe of the instrument into a tedlar bag filled with the calibration gas (generally isobutylene at a concentration of 100 ppm) to calibrate the instrument for medium concentrations. Daily calibration data are recorded in the field log book, including instrument model and serial number.

6.2 FIELD SCREENING

Not applicable.

6.3 SOIL

6.3.1 Surface Soil Sampling

Not applicable.

6.3.2 Subsurface Soil Sampling

Not applicable.

6.4 SEDIMENT SAMPLING

Not applicable.

6.5 WATER SAMPLING

6.5.1 Surface Water Sampling

Not applicable.

6.5.2 Groundwater Sampling

For the borings where grab groundwater samples will be collected a discrete-point sampler with detachable tip (hydropunch sampler) will be used at the bottom of the drill string. Upon attaining the depth where groundwater is expected to be present, the drill string will be pulled back by approximately three feet to allow groundwater to enter the sampler. After allowing time for groundwater to enter the sampler, a small-diameter (1/4-inch O.D.) water-level indicator will be lowered into the boring to verify that groundwater is present. A disposable bailer will then be lowered gently into the boring to collect the groundwater sample. Upon retrieving the bailer from the boring, the sample will be poured into 40-mL sampling vials (pre-preserved at the laboratory with HCl) at a slow, constant rate (100 mL/minute) using a bottom-emptying device. VOC sample vials will be filled completely and immediately capped. The vials will be inverted to ensure that no head space is present. If bubbles appear in a vial (indicating that head space is present), the sample will be discarded and a new sample vial filled, and checked for head space until the required number of VOC sample vials are filled with no head space.

6.5.2.1 Water-Level Measurements

Not applicable.

6.5.2.2 Purging

Not applicable, because the proposed samples are grab samples from temporary borings rather than from developed wells, following standard practice for collection of discrete-point or hydropunch samples from temporary borings.

6.5.2.3 Well Sampling

Not applicable.

6.6 BIOLOGICAL SAMPLING

Not applicable.

6.6.1 Biological Sampling for Chemical Analysis

Not applicable.

6.6.1.1 Fish Samples

Not applicable.

6.6.1.2 Foliage Samples

Not applicable.

6.6.2 Biological Sampling for Species Assessment

Not applicable.

6.7 DECONTAMINATION PROCEDURES

The decontamination procedures that will be followed are in accordance with approved procedures. Decontamination of sampling equipment must be conducted consistently as to assure the quality of samples collected. All equipment that comes into contact with potentially contaminated soil or water will be decontaminated. Disposable equipment intended for one-time use (bailers and rope for lowering bailers) will not be decontaminated, but will be packaged for appropriate disposal. Decontamination will occur prior to and after each use of a piece of reusable equipment.

Large pieces of equipment such as drill rods and discrete-point samplers will be decontaminated by steam-cleaning in a predesignated area on pallets on plastic sheeting. Cleaned equipment will be stored on plastic sheeting in uncontaminated areas. Cleaned small equipment will be stored in plastic bags. Materials to be stored more than a few hours will also be covered.

7.0 SAMPLE CONTAINERS, PRESERVATION AND STORAGE

The number of sample containers, volumes, and materials are listed in Section 5.0. The containers are pre-cleaned and will not be rinsed prior to sample collection. Additionally, the sample containers will be provided pre-preserved with hydrochloric acid by the laboratory vendor.

7.1 SOIL SAMPLES

Not applicable.

7.2 SEDIMENT SAMPLES

Not applicable.

7.3 WATER SAMPLES

Six to eight groundwater samples will be collected for analysis of volatile organic compounds by Method SW8260B. Samples will be collected in 40-mL glass vials pre-preserved with 1:1 hydrochloric acid (HCl) by the vendor. The vials will be filled so that there is no headspace. The samples will be chilled to 4°C immediately upon collection. Three vials of each water sample are required for the laboratory.

7.4 BIOLOGICAL SAMPLES

Not applicable.

7.4.1 Fish Samples

Not applicable.

7.4.2 Foliage Samples

Not applicable.

7.4.3 Biological Sampling for Species Assessment

Not applicable.

8.0 DISPOSAL OF RESIDUAL MATERIALS

In the process of collecting environmental samples the ITSI sampling team will generate different types of potentially contaminated IDW that include the following:

- Used personal protective equipment (PPE)
- Disposable sampling equipment
- Decontamination fluids
- Excess groundwater collected for sample container filling.

The EPA's National Contingency Plan requires that management of investigation derived waste (IDW) generated during sampling comply with all applicable or relevant and appropriate requirements ARARs to the extent practicable. The sampling plan will follow the *Office of Emergency and Remedial Response OERR Directive 9345.3-02* (May 1991), which provides the guidance for the management of IDW. In addition, other legal and practical considerations that may affect the handling of IDW will be considered.

- Used PPE and disposable equipment will be double-bagged and placed in a municipal refuse dumpster. These wastes are not considered hazardous and can be sent to a municipal landfill. Any PPE and disposable equipment that is to be disposed of which can still be reused will be rendered inoperable before disposal in the refuse dumpster.
- Decontamination fluids that will be generated in the sampling event will consist of water, residual contaminants, and water with non-phosphate detergent. The volume and concentration of the decontamination fluid will be sufficiently low to allow disposal at the site or sampling area. The water (and water with detergent) will be poured onto the ground or into a storm drain.
- Purged groundwater will not be generated, because all samples will be grab samples from temporary borings.

9.0 SAMPLE DOCUMENTATION AND SHIPMENT

9.1 FIELD NOTES

9.1.1 Field Logbooks

At a minimum, the following information will be recorded during the collection of each sample:

- Sample location and description
- Site or sampling area sketch showing sample location and measured distances
- Sampler's name(s)
- Date and time of sample collection
- Designation of sample as composite or grab
- Type of sample (soil, sediment or water)
- Type of sampling equipment used
- Field instrument readings and calibration
- Field observations and details related to analysis or integrity of samples (e.g., weather conditions, noticeable odors, colors, etc.)
- Preliminary sample descriptions (e.g., for water: clear water with strong ammonia-like odor)
- Sample preservation
- Lot numbers of the sample containers, sample identification numbers and any explanatory codes, and chain-of-custody form numbers
- Shipping arrangements (overnight air bill number)
- Name of recipient laboratory

In addition to the sampling information, the following specific information will also be recorded in the field logbook for each day of sampling:

- Team members and their responsibilities
- Time of arrival/entry on site and time of site departure
- Other personnel on-site
- Summary of any meetings or discussions with tribal, contractor, or federal agency personnel
- Deviations from sampling plans, site safety plans, and QAPP procedures
- Changes in personnel and responsibilities with reasons for the changes

- Levels of safety protection
- Calibration readings for any equipment used and equipment model and serial number

9.1.2 Photographs

Photographs may be taken at the sampling locations and at other areas of interest in the sampling area. They will serve to verify information entered in the field logbook. For each photograph taken, the following information will be written in the logbook or recorded in a separate field photography log:

- Time, date, location, and weather conditions
- Description of the subject photographed
- Name of person taking the photograph

9.2 LABELING

All samples collected will be labeled in a clear and precise way for proper identification in the field and for tracking in the laboratory. The samples will have pre-assigned, identifiable, and unique numbers. At a minimum, the sample labels will contain the following information: station location, date of collection, analytical parameter(s), and method of preservation. Every sample, including samples collected from a single location but going to separate laboratories, will be assigned a unique sample number.

9.3 SAMPLE CHAIN-OF-CUSTODY FORMS AND CUSTODY SEALS

All sample shipments for analyses will be accompanied by a chain-of-custody record. A copy of the form is found in Appendix B. Form(s) will be completed and sent with the samples for each laboratory and each shipment (i.e., each day). Proper distribution of the forms is found in the "Instructions for Sample Shipping and Documentation" guidance document. If multiple coolers are sent to a single laboratory on a single day, forms will be completed and sent with the samples for each cooler.

The chain-of-custody form will identify the contents of each shipment and maintain the custodial integrity of the samples. Generally, a sample is considered to be in someone's custody if it is either in someone's physical possession, in someone's view, locked up, or kept in a secured area that is restricted to authorized personnel. Until the samples are shipped, the custody of the

samples will be the responsibility of ITSI. The sampling team leader or designee will sign the chain-of-custody form in the "relinquished by" box and note date, time, and airbill number.

The sample numbers for all rinsate samples, reference samples, laboratory QC samples, and duplicates will be documented on this form (see Section 10.0). A copy will be kept for ITSI's master files.

A self-adhesive custody seal will be placed across the lid of each sample. A copy of the seal is found in Appendix B. For VOC samples, the seal will be wrapped around the cap. The shipping containers in which samples are stored (usually a sturdy picnic cooler or ice chest) will be sealed with self-adhesive custody seals any time they are not in someone's possession or view before shipping. All custody seals will be signed and dated.

9.4 PACKAGING AND SHIPMENT

All sample containers will be placed in a strong-outside shipping container (a steel-belted cooler). The following outlines the packaging procedures that will be followed for low-concentration samples.

1. When ice is used, pack it in zip-locked, double plastic bags. Seal the drain plug of the cooler with fiberglass tape to prevent melting ice from leaking out of the cooler.
2. The bottom of the cooler should be lined with bubble wrap to prevent breakage during shipment.
3. Check screw caps for tightness and, if not full, mark the sample volume level of liquid samples on the outside of the sample bottles with indelible ink.
4. Secure bottle/container tops with clear tape and custody seal all container tops.
5. Affix sample labels onto the containers with clear tape.
6. Wrap all glass sample containers in bubble wrap to prevent breakage.
7. Seal all sample containers in heavy-duty plastic zip-lock bags. Write the sample numbers on the outside of the plastic bags with indelible ink.
8. Place samples in a sturdy cooler lined with a large plastic trash bag. Enclose the appropriate COC(s) in a zip-lock plastic bag affixed to the underside of the cooler lid.
9. Fill empty space in the cooler with bubble wrap or Styrofoam peanuts to prevent movement and breakage during shipment. Vermiculite should also be placed in the cooler to absorb spills if they occur.

10. Ice used to cool samples will be double-sealed in two zip-lock plastic bags and placed on top and around the samples to chill them to the correct temperature.
11. Each ice chest will be securely taped shut with fiberglass strapping tape, and custody seals will be affixed to the front, right and back of each cooler.

Records will be maintained by ITSI's sample custodian of the following information:

- Name and location of the site or sampling area
- Project number
- Total number(s) by estimated concentration and matrix of samples
- shipped to laboratory
- Carrier, airbill number(s), method of shipment (priority next day)
- Shipment date and when it should be received by lab
- Irregularities or anticipated problems associated with the samples
- Whether additional samples will be shipped or if this is the last shipment.

10.0 QUALITY CONTROL

10.1 FIELD QUALITY CONTROL SAMPLES

10.1.1 Assessment of Field Contamination (Blanks)

10.1.1.1 Equipment Blanks

Not applicable - equipment rinsate blanks will be not be collected because disposable sampling equipment will be used.

10.1.1.2 Field Blanks

Not applicable.

10.1.1.3 Trip Blanks

Trip blanks will be prepared to evaluate if the shipping and handling procedures are introducing contaminants into the samples, and if cross-contamination in the form of VOC migration has occurred between the collected samples. A minimum of one trip blank will be submitted to the laboratory for analysis with every shipment of samples for VOC analysis. Trip blanks are 40-mL vials that have been filled with HPLC-grade water that has been purged so it is VOC-free and shipped with the empty sampling containers to the site or sampling area prior to sampling. The sealed trip blanks are not opened in the field and are shipped to the laboratory in the same cooler with the samples collected for volatile analysis. The trip blanks will be packaged and sealed in the manner described for the environmental samples. A separate sample number will be assigned to each trip sample and it will be submitted blind to the laboratory.

10.1.1.4 Temperature Blanks

For each cooler that is shipped or transported to an analytical laboratory a 40-mL VOA vial will be included that is marked "temperature blank." This blank will be used by the sample custodian to check the temperature of samples upon receipt.

10.1.2 Assessment of Field Variability (Field Duplicate Samples)

A field duplicate water sample will be collected from a boring within the probable footprint of the plume. The duplicate sample will be collected because groundwater is suspected of exhibiting moderate concentrations of contaminants, based on contamination patterns during the most-recent sampling event. The duplicate sample will be labeled GW-9.

To collect the duplicate water samples, alternate the filling of the two sets of VOA bottles. Duplicate samples will be packaged and sealed in the same manner as other samples of the same matrix. A separate sample number will be assigned to each duplicate, and it will be submitted blind to the laboratory.

10.2 BACKGROUND SAMPLES

Not applicable.

10.3 FIELD SCREENING AND CONFIRMATION SAMPLES

Not applicable.

10.3.1 Field Screening Samples

Not applicable.

10.3.2 Confirmation Samples

Not applicable.

10.3.3 Split Samples

Not applicable.

10.4 LABORATORY QUALITY CONTROL SAMPLES

For water samples, double volumes of samples are supplied to the laboratory for its use for QC purposes (matrix spike/matrix spike duplicate samples). Two sets of water sample containers are filled and all containers are labeled with a single sample number. For VOC samples this would result in 6 vials being collected instead of 3.

The laboratory will be alerted as to which sample is to be used for QC analysis by a notation ("MS/MSD") on the sample container label and the chain-of-custody record or packing list.

At a minimum, one laboratory QC sample is required per 14 days or one per 20 samples (including blanks and duplicates), whichever is greater. If the sample event lasts longer than 14 days or involves collection of more than 20 samples per matrix, additional QC samples will be designated.

For this sampling event, a groundwater sample will be collected from a boring placed within the probable footprint of the plume and designated a laboratory QC sample (using the abbreviation MS/MSD). A matrix spike/matrix spike duplicate sample will be collected from this location because groundwater is suspected of exhibiting moderate concentrations of contaminants, based on historic sampling results.

11.0 FIELD VARIANCES

As conditions in the field may vary, it may become necessary to implement minor modifications this sampling and analysis plan. When appropriate, the QA Office will be notified and a verbal approval will be obtained before implementing the changes. Modifications to the approved plan will be documented in the sampling project report.

12.0 REFERENCES

Golder Associates (Golder), 1996. *Report on Soil and Groundwater Investigation, Anchor Glass Container Corporation, Plant 32 Facility*. May 9.

Regional Water Quality Control Board San Francisco Bay Region (RWQCB), Groundwater Committee (1999). *East Bay Plain Groundwater Basin, Beneficial Use Evaluation Report, Alameda and Contra Costa Counties, CA*. June.

Regional Water Quality Control Board (RWQCB), 1996. *Concurrence With Your August 21, 1995 Proposal to Abandon On-Site Monitoring Wells at 21 & 24 Cannery Court, Hayward*. January 16.

Regional Water Quality Control Board (RWQCB), 1995. *Chlorinated Solvent Issue at 21 & 24 Cannery Court, Hayward, California*. July 21.

U.S. Environmental Protection Agency (EPA), 1994. Contract Laboratory Program: National Functional Guidelines for Organic Data Review. EPA-540/R-94/012. February.

Tables

Table 3-1

**Quality Assurance Goals: Accuracy, Percent Recovery
Limited Phase II Targeted Brownfields Assessment
Cannery Redevelopment Area, City of Hayward**

Method and Parameters	Water Samples			Soil Samples		
	LCS Recovery	MS/MSD Recovery	Surrogate Recovery (a)	LCS Recovery	MS/MSD Recovery	Surrogate Recovery (a)
EPA Method 8260B: Volatile Organic Compounds						
Benzene	76-127	76-127	NA	NA	NA	NA
Chlorobenzene	75-130	75-130	NA	NA	NA	NA
1,1-Dichloroethene	61-145	61-145	NA	NA	NA	NA
Toluene	76-125	76-125	NA	NA	NA	NA
Trichloroethene	71-120	71-120	NA	NA	NA	NA
4-Bromofluorobenzene	NA	NA	86-115	NA	NA	NA
1,2-Dichloroethane-d4	NA	NA	80-120	NA	NA	NA
Toluene-d8	NA	NA	88-110	NA	NA	NA
Dibromofluoromethane	NA	NA	86-118	NA	NA	NA

(a) Other surrogate compounds may be substituted, if appropriate.
NA Not applicable.

Table 3-2

**Quality Assurance Goals: Precision, Relative Percent Difference
Limited Phase II Targeted Brownfields Assessment
Cannery Redevelopment Area, City of Hayward**

Method and Parameters	Water Samples		Soil Samples	
	MS/MSD RPD	Field Duplicate RPD(a)	MS/MSD RPD	Field Duplicate RPD(a)
EPA Method 8260B Volatile Organic Compounds				
Benzene	11	50	NA	NA
Chlorobenzene	13	50	NA	NA
1,1-Dichloroethene	14	50	NA	NA
Toluene	13	50	NA	NA

(a) Field duplicate RPD applies to all target analytes in the test method.
NA Not applicable.

Table 3-3

**Analytical Methods, Parameters for Analysis, and Practical Quantitation Limits
Limited Phase II Targeted Brownfields Assessment
Cannery Redevelopment Area, City of Hayward**

Method and Parameters	Practical Quantitation Limit	
	Water ($\mu\text{g/L}$)	Soil ($\mu\text{g/kg}$)
EPA Method 8260B: Volatile Organic Compounds		
Acetone	20	NA
Benzene	5.0	NA
Bromodichloromethane	5.0	NA
Bromoform	5.0	NA
Bromomethane	10	NA
2-Butanone	20	NA
Carbon disulfide	5.0	NA
Carbon tetrachloride	5.0	NA
Chlorobenzene	5.0	NA
Chloroethane	10	NA
Chloroform	5.0	NA
Chloromethane	10	NA
Dibromochloromethane	5.0	NA
1,1-Dichloroethane	5.0	NA
1,2-Dichloroethane	5.0	NA
1,1-Dichloroethene	5.0	NA
1,2-Dichloroethene (total)	5.0	NA
1,2-Dichloropropane	5.0	NA
cis-1,3-Dichloropropene	5.0	NA
trans-1,3-Dichloropropene	5.0	NA
Ethylbenzene	5.0	NA
2-Hexanone	20	NA
4-Methyl-2-pentanone	20	NA
Methylene chloride	10	NA
Styrene	5.0	NA
1,1,1,2-Tetrachloroethane	5.0	NA

Table 3-3 (continued)

**Analytical Methods, Parameters for Analysis, and Practical Quantitation Limits
Limited Phase II Targeted Brownfields Assessment
Cannery Redevelopment Area, City of Hayward**

Method and Parameters	Practical Quantitation Limit	
	Water ($\mu\text{g/L}$)	Soil ($\mu\text{g/kg}$)
EPA Method 8260B: Volatile Organic Compounds		
Tetrachloroethene	5.0	NA
Toluene	5.0	NA
1,1,1-Trichloroethane	5.0	NA
1,1,2-Trichloroethane	5.0	NA
1,1,2-Trichloro-1,2,2-trifluoroethane	5.0	NA
Trichloroethene	5.0	NA
Vinyl acetate	10	NA
Vinyl chloride	10	NA
Xylenes (total)	5.0	NA

Table 3-4

**Summary of Corrective Actions
Limited Phase II Targeted Brownfields Assessment
Cannery Redevelopment Area, City of Hayward**

QC Parameter Out of Control	Corrective Action
Sample Handling (includes preservation and storage temperature)	<ol style="list-style-type: none"> 1. Do not proceed with analysis. 2. Collect new samples.
Holding Times	<ol style="list-style-type: none"> 1. Do not proceed with analysis. 2. Collect new samples.
Initial Calibration	<ol style="list-style-type: none"> 1. Evaluate system. 2. Recalibrate as necessary. 3. Analyze samples only after initial calibration is acceptable.
Continuing Calibration	<ol style="list-style-type: none"> 1. Evaluate system. 2. Reanalyze standard. 3. Recalibrate as necessary. 4. Reanalyze affected samples.
Method Blank	<ol style="list-style-type: none"> 1. Evaluate system. 2. Reextract and reanalyze method blank and associated samples. 3. Analyze samples only after method blank is acceptable.
LCS recovery	<ol style="list-style-type: none"> 1. Evaluate system. 2. Reextract and reanalyze LCS and associated samples within the holding time. 3. Report sample data only after LCS is acceptable.
Surrogate recovery	<ol style="list-style-type: none"> 1. Evaluate system. 2. Reanalyze sample within the holding time. If acceptable, report acceptable data only. 3. If unacceptable, attempt to reextract and reanalyze the sample within the holding time (expiration of holding time does not remove the need to reextract and reanalyze the sample). 4. If no control exceedance is observed and the reanalysis is within the holding time, report acceptable data for sample and surrogate. 5. If a control exceedance is observed, or if reanalysis not within the holding time, report both sets of sample and surrogate data.

Table 3-4 (Continued)

**Summary of Corrective Actions
Limited Phase II Targeted Brownfields Assessment
Cannery Redevelopment Area, City of Hayward**

QC Parameter Out of Control	Corrective Action
Internal standard recovery	<ol style="list-style-type: none"> 1. Evaluate system. 2. Reanalyze sample within the holding time. If acceptable, report acceptable data only. 3. If unacceptable, attempt to reextract and reanalyze the sample within the holding time (expiration of holding time does not remove the need to reextract and reanalyze the sample). 4. If no control exceedance is observed and the reanalysis is within the holding time, report acceptable data for sample and internal standards. 5. If a control exceedance is observed, or if reanalysis not within the holding time, report both sets of sample and internal standard data.
MS/MSD recovery and RPD	<ol style="list-style-type: none"> 1. Evaluate system. 2. Reanalyze MS/MSD. If acceptable, report acceptable data only. 3. If unacceptable, reextract and reanalyze MS/MSD and report both sets of MS/MSD data.
Matrix Duplicate RPD	<ol style="list-style-type: none"> 1. Evaluate system. 2. Reanalyze matrix duplicate. If acceptable, report acceptable data only. 3. If unacceptable, reextract and reanalyze matrix duplicate and report both sets of matrix duplicate data.
Field-generated Blanks (includes trip blanks, equipment blanks, and field water blanks)	<ol style="list-style-type: none"> 1. Evaluate method blank. 2. Evaluate field sampling and decontamination procedures. 3. Evaluate field water source. 4. Modify sampling and decontamination procedures, as appropriate.

Table 3-5

**Summary of Calibration Procedures
Limited Phase II Targeted Brownfields Assessment
Cannery Redevelopment Area, City of Hayward**

Method Number	Parameters	Calibration	Frequency	Acceptance Criteria	Corrective Action
8260B	Volatile Organic Compounds	Check of instrument tuning	Every 12 hours	Refer to method	(1) Retune instrument (2) Reanalyze tuning check
		Multipoint calibration (minimum of 5 points)	Initially and as required	$\%RSD \leq 30\%$ for CCC $RF \geq 0.10$ for SPCC	(1) Evaluate system (2) Recalibrate
		Method blanks and instrument blanks	After initial calibration	No target analytes present above half of the PQL	(1) Reanalyze blank (2) Clean system (3) Reanalyze affected samples
		Continuing calibration check standard	Every 12 hours	80-120% recovery for CCC $RF \geq 0.10$ for SPCC	(1) Evaluate system (2) Reanalyze standard (3) Recalibrate (4) Reanalyze affected samples

CCC Continuing calibration compounds

RSD Relative standard deviation of response factors

RF Response factor

SPCCs System performance check compounds

Table 3-6

**Data Validation Procedures
Limited Phase II Targeted Brownfields Assessment
Cannery Redevelopment Area, City of Hayward**

Control Parameter	Exceedance of Control Limits	Qualification of Detected Results	Qualification of Nondetected Results	Associated Results
Holding Times	Holding time exceeded by 1 - 7 days	J-	J-	All method results in affected sample
	Holding time exceeded by > 7 days	J-	R	All method results in affected sample
Laboratory Blanks	Analyte detected in blank. Determine action level, x10 for common lab contaminant or x5 for other analytes	U detected results below action level	Not qualified	Analyte results in the analytical batch
Trip Blanks	Same as laboratory blanks	U detected results below action level	Not qualified	Analyte results in samples in the same cooler
Equipment Blanks	Same as laboratory blanks	U detected results below action level	Not qualified	Analyte results in samples in the same sampling event
Surrogate Recovery	recovery > UCL	J+	Not qualified	All method results in affected sample
	LCL > recovery > 10%	J-	J-	
	recovery < 10%	J-	R	
LCS Recovery	recovery > UCL	J+	Not qualified	Analyte results in the analytical batch or sub-analytical batch
	LCL > recovery > 10%	J-	J-	
	recovery < 10%	J-	R	
MS/MSD Recoveries	recovery > UCL	J+	Not qualified	Analyte results in the spiked sample.
	LCL > recovery > 10%	J-	J-	
	recovery < 10%	J-	R	
MS/MSD RPD	RPD > CL	J	J	Analyte results in the spiked sample.

Table 3-6 (continued)

**Data Validation Procedures
Limited Phase II Targeted Brownfields Assessment
Cannery Redevelopment Area, City of Hayward**

Control Parameter	Exceedance of Control Limits	Qualification of Detected Results	Qualification of Nondetected Results	Associated Results
Initial Calibration %RSD	%RSD > CL or $r < 0.995$	J	J	Analyte results associated with the initial calibration
Initial Calibration RRF	RRF < 0.05	J	R	Analyte results associated with the initial calibration
Continuing Calibration %D	%D > UCL -50% < %D < LCL %D < -50%	J+ J- J-	Not qualified J- R	Analyte results associated with the continuing calibration
Field Duplicate RPD	RPD > CL	Not qualified; discuss in report	Not qualified; discuss in report	Analyte results in the sample and duplicate
Internal Standard Area Counts	Area counts > 2x the associated standard	J+	Not qualified	Associated analyte results in the sample
	Area counts < 0.5x the associated standard	J-	R	Associated analyte results in the sample

Table 5-1

**Request for Analytical Services Matrix – Groundwater
Limited Phase II Targeted Brownfields Assessment
Cannery Redevelopment Area, City of Hayward**

ANALYSES REQUESTED							Contract Laboratory Program Analytical Services (CLPAS)
CHEMISTRY TYPE							Organics
SPECIFIC ANALYSES REQUESTED							VOCs
PRESERVATIVES							Add 1:1 HCl to pH <2 Chill to 4°C
ANALYTICAL HOLDING TIME(S)							Hold <14 days
CONTRACT HOLDING TIMES(S)							Hold <14 days
NUMBER OF SAMPLES x NUMBER OF SAMPLE CONTAINERS							No. of Containers per Analysis
Sample Number	Sample Location	Sample Depth	Sampling Date	Special Designation	Concentration		
					LOW	MED	
GW-1					X		3 x 40 mL glass vials
GW-2					X		3 x 40 mL glass vials
GW-3					X		3 x 40 mL glass vials
GW-4					X		3 x 40 mL glass vials
GW-5					X		3 x 40 mL glass vials
GW-6				+ MS/MSD	X		6 x 40 mL glass vials
GW-7 (Alt)					X		3 x 40 mL glass vials
GW-8 (Alt)					X		3 x 40 mL glass vials
GW-9 (dup)					X		3 x 40 mL glass vials
TOTAL					6-8		

Figures

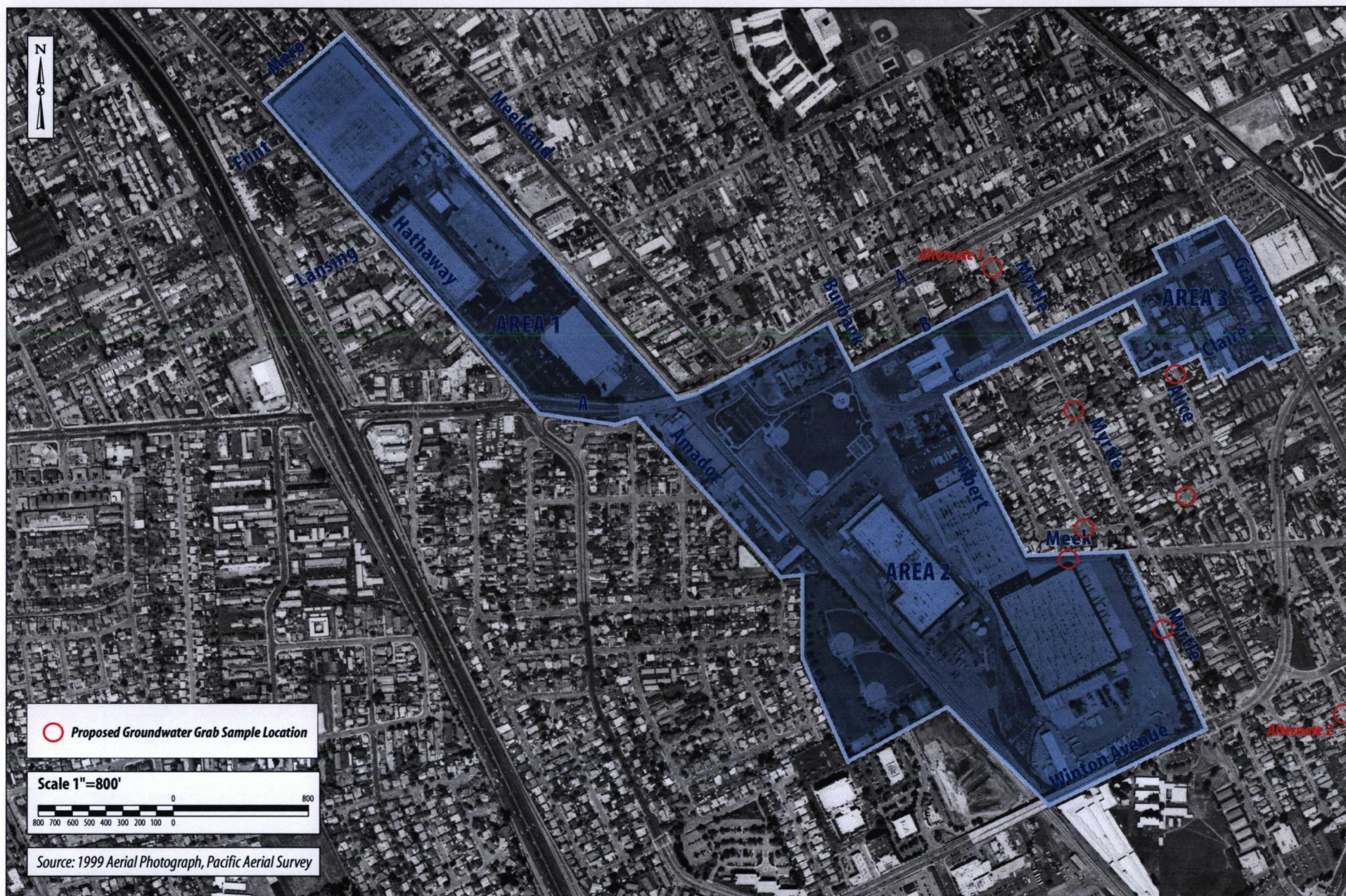


Figure 1—Approximate Locations of Proposed Groundwater Grab Samples
 Phase I Targeted Brownfields Assessment
 Cannery Redevelopment Area, City of Hayward

Prepared For:



United States
Environmental Protection
Agency



U.S. Army
Corps of Engineers

Prepared By:



Innovative
Technical
Solutions, Inc.

APPENDIX A

**STANDARD OPERATING PROCEDURE FOR
METHOD SW8260B**

CHROMALAB, INC.

Environmental Service (SDB)

STANDARD OPERATING PROCEDURES

COMPANY CONFIDENTIAL AND PROPRIETARY

Volatile Organics in Soil/Water
Eff. Date: 08/11/00

Approved By: *Eric Tam*

SOP ID: 06.06A Rev 01

CONTROLLED DISTRIBUTION

SOP #06.06A
Rev 01

Volatile Organics in Soil/Water/Air

REFERENCE: EPA SW846 Method 8260B, December 1996

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Full Signature Approvals Are Kept on File
In The Quality Assurance Department

Approved By: _____

Technical Reviewer

Date: ____/____/____

Approved By: _____

Quality Assurance

Date: ____/____/____

Approved By: _____

Laboratory Director

Date: ____/____/____

STANDARD OPERATING PROCEDURES

COMPANY CONFIDENTIAL AND PROPRIETARY

Volatile Organics in Soil/Water

SOP ID: 06.06A Rev 01

Eff. Date: 08/11/00

Approved By: *Eric Tam***1.0 SCOPE and APPLICATION**

- 1.1 Method 8260B is designed to determine the concentration of volatile organic compounds on the Target Analyte List (TAL) from aqueous, sediment, soil and air samples from hazardous waste sites.

2.0 SUMMARY

- 2.1 Volatile compounds are introduced by purge-and-trap into a gas chromatograph. Helium is bubbled through a 10 ml aqueous sample (for low level solid samples, a mixture of 5g sample and reagent water) contained in a specifically designed purging chamber at elevated temperatures.* The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is complete, the sorbent column is heated and backflushed with helium to desorb the purgeables onto the gas chromatograph column. The gas chromatograph is temperature programmed to separate the purgeables which are then detected by the mass spectrometer.

* Only soil samples use a heated purge from Archon and Precept II.

- 2.2 Solid samples –

- 2.2.1 Low level - Helium is bubbled through a mixture of a 5g sample and reagent water contained in a specifically designed purging chamber at elevated temperatures. Medium level - 4 g of soil is extracted with 10 ml of methanol. A portion of the methanol extract is diluted to 10 ml with reagent water. The sample extract water is analyzed as a water.

3.0 SAFETY

- 3.1 Laboratory coats, safety glasses and protective gloves must be worn.

4.0 INTERFERENCE'S

- 4.1 Impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be demonstrated to be free of contamination under the conditions of the analysis by running laboratory reagent blanks.
- 4.2 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. To reduce carry-over, the purging device and syringe must be rinsed with reagent water between sample analysis. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of a reagent blank to check for cross contamination. For samples containing large amounts of water soluble materials, suspended solids, high boiling compounds or high purgeable levels, between analysis, it may be necessary to wash out the purging device and then let it dry at 105°C. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.
- 4.3 The laboratory where volatiles analysis is performed should be completely free of solvents other than methanol.
- 4.4 Problems have been associated with the following compounds analyzed by this method:
- 4.4.1 Chloromethane, vinyl chloride, bromomethane, chloroethane can display peak broadening if the compounds are not delivered to the GC column in a tight band.
- 4.4.2 Acetone, 2-Butanone (MEK), Carbon Disulfide, 1,2-Dibromo-3-Chloropropane, 2-Hexanone, and 4-Methyl-2-Pentanone (MIBK) have poor purge (P.P.) efficiencies.
- 4.4.3 1,1,1-Trichloroethane and all dichloroethane can dehydrogenate during storage or analysis.

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- 4.4.4 Tetrachloroethane and 1,1-dichloroethane can be degraded by contaminated transfer lines in the purge and trap systems and/or active sites in trapping materials.
- 4.4.5 Chloromethane can be lost if the purge flow is too fast.
- 4.4.6 Bromoform is one of the compounds most likely to be adversely affected by the cold spots and/or active sites in the transfer lines. Response of its quantitation ion (m/z 173) is directly affected by the tuning of the GC/MS to meet the instrument performance criteria for BFB at ions 174/176. Increasing the m/z 174/176 ratio may improve bromoform response.

5.0 SAMPLE COLLECTION, PRESERVATION, and HANDLING

- 5.1 All solid samples shall be collected in a clear wide mouth jar, brass tube, or preserved soil vials for purge-and trap, depending on the preparation method used. All aqueous samples shall be collected in 40 ml VOA vials and preserved with HCl to pH 2 or lower.
- 5.2 The samples must be protected from light and refrigerated at 4°C from the time of receipt until 30 days after delivery of the sample to the lab. After 30 days, the sample may be disposed of in a manner that complies with all applicable regulations.
- 5.3 The sample must be stored in an atmosphere demonstrated to be free of all potential contaminants below the reporting limit of samples.
- 5.4 Standards and samples must be stored separately.
- 5.5 Soil and sediment samples must be analyzed within 14 days from the sampling date. Aqueous samples must be analyzed within 14 days from the sampling date.

6.0 EQUIPMENT and REAGENTS**6.1 Equipment**

- 6.1.1 Gas tight Micro syringes - 10 ul and larger, 0.006 inch ID needle.
- 6.1.2 Syringe valve - two way, with Luer ends, if applicable to the purging device.
- 6.1.3 Syringe - 5 ml & 10 ml gas tight.
- 6.1.4 40 ml VOA vial
- 6.1.5 Balance - analytical, capable of accurately weighing 0.0001 g, and a top loading balance capable of weighing 0.1 g.
- 6.1.6 Spatula
- 6.1.7 Volumetric flasks - class A with ground glass stoppers.
- 6.1.8 Sprayers
- 6.1.9 Purge and trap device - consists of three separate pieces of equipment: The sample purger, trap, and the desorber. The Tekmar Precept II / LSC 3000 / LSC 2000, ALS 2016, and Varian Archon autosampler are used at this lab. Also a Tekmar Precept II or Varian Archon with heated purge is used for all soil QC and soil samples. DB-VRX columns (60m x 0.25mm x 1.4µm) or equivalent will be used.
- The sample purger must be designed to accept 5-10 ml sample with a water column at least 3 cm deep. The gaseous head space between the water and the trap must have a total volume of less than 15 ml. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column.
 - A purge trap C or K is used and must be 25 cm long and have an inside diameter of at least 0.105 inch. The trap must be packed to contain the minimum lengths of

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absorbents: 15 cm of Tenax - GC (60/80 mesh), 8 cm of silica gel (35/60 mesh, grade 15, or equivalent), 1 cm charcoal.

- The desorber should be capable of rapidly heating the trap to 200°C. The polymer section of the trap should not be heated higher than 200°C and the remaining section should not exceed 230°C during the bakeout mode.
- A heater capable of maintaining the purge device at 40°C is to be used.

6.1.10 GC/MS system

- Gas Chromatograph - the gas chromatograph (GC) system must be capable of temperature programming and have a flow controller that maintains a constant column flow rate throughout desorption and temperature program operations. The system must include or be interfaced to a purge and trap system as specified in paragraph 6.1.7 and have all required accessories. HP 5890A GC and Saturn GC/MS are used. The HP #1 chromatograph is interfaced with cryogenic cooling which maintains temperature at 30°C or lower under a stream of liquid nitrogen. HP #2 and Saturn do not use cryogenic cooling. HP #2 has a starting temperature of 45°C.

- Mass Spectrometer - must be capable of scanning from 35 to 260 amu every 1 second or less, utilizing 70 electron volts energy in the electron impact ionization mode, and producing mass spectrum which meets all the instrument performance acceptance criteria when 50 ng of BFB is injected into the GC. The purge and trap GC/MS system must be in a room that is demonstrated to be free of all potential contaminants which will interfere with the analysis. HP 5972 GC/MS and Saturn GC/MS are used.

Electron Energy: 70 eV
Mass Range: 35-260 amu
Scan Time: 0.6 - 2 sec/scan

- GC/MS interface - any GC to MS interface that gives acceptable calibration points, at 50 ng or less per injection, for each of the parameters of interest and achieves all acceptance criteria may be used. GC to MS interfaces constructed of all glass-lined materials are recommended. Glass can be deactivated by silanization.
- Data system - a computer must be interfaced to the mass spectrometer that allows the continuous acquisition and storage, on machine readable media, of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of specified mass and plotting such ions abundance's versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integration of the abundance in any EICP between specified time or scan number limits. Also, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra.
- Magnetic tape storage device and/or equivalent (e.g. optical disk) - must be capable of recording data and must be suitable for long term, off line storage.

6.2 Reagents

- 6.2.1 Reagent water - defined as water in which an interferant is not observed at or above the PQL of the parameter of interest; filtered tap water.
- 6.2.2 Methanol - purge and trap grade or equivalent.

7.0 **SAMPLE PREPARATION**

STANDARD OPERATING PROCEDURES

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Volatile Organics in Soil/Water

SOP ID: 06.06A Rev 01

Eff. Date: 08/11/00

Approved By: *Eric Tam*

- 7.1 Soil Samples - Method 5035
- 7.2 Aqueous Samples - Method 5030B

8.0 STANDARDS

- 8.1 Stock Standards expire in 6 months except gases which expire in two months.
- 8.2 Secondary Dilution Standards
 - 8.2.1 Using stock standard solutions, prepare secondary dilution standards in methanol that contain the compounds of interest, either singularly or mixed together. Secondary dilution solutions should be prepared at concentrations that can be easily diluted to prepare working solutions.
 - 8.2.2 Prepare fresh secondary dilution standards for gases and for reactive compounds such as styrene every week or sooner, if standard has degraded or evaporated. Secondary dilution standards for the other purgeable compounds must be replaced after one week, or sooner if the standard has degraded or evaporated. It may be necessary to replace the standards more frequently if either check exceeds a 20% drift.
- 8.3 Instrument Performance Check Solution - Bromofluorobenzene (BFB)
 - 8.3.1 Prepare a 25 ng/ul solution of BFB in methanol (inject 2 uL). Prepare fresh BFB solution every six months, or sooner, if the solution degraded or evaporated.
- 8.4 Calibration Standard Solution
 - 8.4.1 Prepare a working calibration standard at 200 ug/ml containing all of the purgeable target compounds in methanol. Prepare fresh working standards as needed.
- 8.5 Internal Standard and Surrogate Spiking Solution
 - 8.5.1 Prepare an internal standard spiking solution containing 1,4-Dichlorobenzene-d4, Chlorobenzene-d5, and 1,4-Difluorobenzene and Pentafluorobenzene at 50 ug/ml
 - 8.5.2 Prepare a surrogate spiking solution containing Toluene-d8, 1,2-dichloroethane-d4, Dibromofluoromethane, and p-Bromofluorobenzene at 100 ug/mL. Add 5 uL into 10 mL of sample to give a final concentration at 500 ng. Prepare new standards as needed.
- 8.6 Volatile Matrix Standard Spiking Solution
 - 8.6.1 Prepare a spiking solution in methanol that contains the following compounds at 100 ug/mL: 1,1-Dichloroethene, Trichloroethene, Chlorobenzene, Toluene, and Benzene. All samples analyzed following Navy guidance document must be spiked with a matrix spike solution containing all analytes of interest. All compounds spiked must be reported.
 - 8.6.2 Matrix spikes also serve as duplicates; therefore, add a 5 uL aliquot of this solution to each of two portions from one sample chosen for spiking.
- 8.7 Initial Calibration Standard Solutions
 - 8.7.1 A minimum of five levels of initial calibration standards should be prepared containing all of the purgeable target compounds and system monitoring compounds at concentrations within working range of the GC/MS system. One level must be prepared to meet project requirements. With every initial calibration, an independent check standard must be analyzed at a concentration midpoint of the ICAL. All compounds must agree within the specified CCC and SPCC requirement listed in section 9.4.2.5 for CCV acceptance. The

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second source check must meet the same requirements as the CCV or the initial calibration is not considered acceptable. The surrogate concentration must be injected at 5 levels for the initial calibration. There are several analytes which demonstrate poor purging efficiency. These compounds are designated in 4.4.2. The analyst may choose to add a sixth or seventh concentration for the compounds listed as (P.P.) since the spectra and response may not be adequate at the L1/L2 levels. The calibration must also meet all the requirements of section 9.4 before any samples may be analyzed.

- 8.7.2 The calibration standards are prepared in the 10.0 mL syringe used to inject the standard into the purging device:

Syringe - remove the plunger from the 10 mL syringe. Pour reagent water into the syringe barrel to just short of overflowing. Replace the plunger and compress the water. Invert the syringe and vent the residual air. Adjust the water to 10.0 mL, the calibration standard is to be added.

8.8 Storage of Standards

- 8.8.1 Store the stock standards in a Teflon sealed screw-cap bottles with minimal headspace at -10 to -20°C. Protect the standards from light. Once the bottles containing the stock standard solution has been opened, it may be used for no longer than 6 months. *For gases (BP < 30 °C) the expiration is 2 months after the ampule is opened.*
- 8.8.2 Store secondary dilution standards in Teflon screw-cap bottles with minimal headspace at -10 to -20°C. Protect them from light. The secondary dilution standards must be checked frequently for signs of degradation or evaporation, especially just prior to preparing the working calibration standards from them.
- 8.8.3 Gas standards must be discarded after one hour unless they are set up to be purged in the autosampler.
- 8.8.4 Purgeable standards must be stored separately from other standards. Expired volatiles standards used for non-quantitation purposes will be labeled as such and stored in a designated area in the refrigerator.

9.0 ANALYTICAL PROCEDURE

9.1 Recommended Instrument Operation Conditions - SATURN III

9.1.1 Purge and Trap Device Analytical Conditions

Purge Conditions:	Saturn 3:
Purge Gas:	Helium
Purge Time:	8.0 min
Purge Temperature	Water - ambient Low Level Soils- 40°C
Desorb Preheat:	155°C
Desorb Temperature:	205°C
Desorb Time:	1.0 min (0.90 soils)
Bake time:	10 min @ 180°C

Before initial use, condition the trap 30 minutes at approximately 230°C by backflushing with at least 20 ml/min flow of inert gas. Vent the trap effluent to the room and not to the analytical column.

9.2 Recommended Gas Chromatograph

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Instrument	Saturn GC/MS 3
Initial Temp: °C	45
Initial Hold time:	10.00
Ramp Rate: °C/Min.	12.0
Final Temperature: °C	190
Time: Min.	12.08
Hold Time: Min.	2.00
Ramp Rate: °C/Min.	6.0
Final Temperature: °C	240
Time: Min.	8.33
Final Hold Time:	Until target compounds

The columns used are RTX-624, HP-624, or J&W DB-624 (60m x 0.32mm x 1.8 micron).

9.3 Mass Spectrometer

9.3.1 The following are the required mass spectrometer conditions:

Electron Energy: 70 eVolts
Mass Range: 35-260 amu
Scan Time: 0.6-2 sec/scan

9.3.2 The GC/MS system must be tuned to meet the BFB tuning criteria prior to the analysis of any samples or standards.

9.3.3 The analysis of the instrument performance solution may be performed as follows:

- As an injection of up to 50 ng of BFB into the GC/MS or,
- By adding 50 ng of BFB to 10 ml of reagent water and analyzing the resulting solution as if it were an environmental sample.
- BFB may not be analyzed simultaneously with the calibration standard.

9.3.4 The mass spectrum of BFB must be acquired in the following manner: The average of a minimum of 3 scans. Background subtraction is required and may be accomplished by using a single scan no more than 20 scans prior to the elution of BFB. The subtraction of any part of the BFB peak is not acceptable. Alternately, other documented approaches by the instrument manufacturer may be used.

9.3.5 The analysis of the instrument performance check solution must meet the ion abundance criteria given below:

Table I: BFB KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	15-40 % of mass 95
75	30-60 % of mass 95
95	base peak, 100 % relative abundance
96	5-9 % of mass 95
173	less than 2 % of mass 174
174	greater than 50 % of mass 95
175	5-9 % of mass 174
176	greater than 95% but less than 101% of mass 174
177	5-9 % of mass 176

If the tune exceeds any of the criteria in table #1 or the visual inspection, the tune must be considered out of control and corrective action taken.

9.3.6 The instrument performance check solution must be injected once at the beginning of each 12 hour period, during which samples or standards are to be analyzed. The time period in

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which all sample, blanks, standards, and spikes are analyzed starts at the injection time of the performance check solution of BFB. The time period ends after the 12 hours has elapsed according to the system clock.

9.4 Calibration

9.4.1 Prior to the analysis of samples and required blanks, and after the instrument performance check solution criteria have been met, each GC/MS systems must be calibrated at a minimum of 5 concentrations to determine the sensitivity and linearity of GC/MS response for the purgeable target compounds. All compounds reported must be calibrated using 5 points. *USACE requires that the low standard from the initial calibration curve be used as the practical quantitation limit for the method.*

9.4.2 Internal Standard Calibration Procedure. Internal standard is to be injected into all of the 5 point calibration standards to have a final concentration of 25 µg/L. The internal standards are as follows: 1,4-Dichlorobenzene-d4, 1,4-Difluorobenzene, and Chlorobenzene-d5 and Pentafluorobenzene. This must be done for both water and low soil calibration (unheated vs. heated purge). Extracts of medium soils may be analyzed using the calibration for water or soil samples.

9.4.2.1 Prepare calibration standards at a minimum of 5 concentration levels for each of the target compound and system monitoring compounds, as specified in section 8.7.

9.4.2.2 Prepare a spiking solution containing each of the internal standards using the procedure described in paragraph 8.6.

9.4.2.3 Analyze each calibration standard, according to paragraph 9.4.1, adding 5 µl of the internal standard spiking solution directly into the syringe. Tabulate the area response of the characteristic ions in the extracted ion profile (EICP) against the concentration for each compound and internal standard and calculate the relative response factors (RRF) for each compound using Equation.

$$RRF = \frac{A_x \times C_{is}}{A_{is} \times C_x}$$

where: A_x = Area of the EICP for the compound to be measured
 A_{is} = Area of the EICP for the specific internal standard.
 C_{is} = Concentration of the internal standard
 C_x = Concentration of the compound to be measured

9.4.2.4 The average RF must be calculated for all compounds. Calculate the % Relative Standard Deviation (%RSD) of the RRF values over the working range of the curve.

$$\% RSD = \frac{\text{Standard Deviation}}{\text{Mean}} \times 100$$

9.4.2.5 The curve is considered acceptable if the Calibration Check Compounds (CCC's) and the System Performance Check Compounds (SPCC's) are in compliance with the following:

CCC'sMaximum %RSD Maximum %Diff

Vinyl Chloride

≤30

(Daily Calibration Check)

≤20

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1,1-Dichloroethene	≤30	≤20
Chloroform	≤30	≤20
1,2-Dichloropropane	≤30	≤20
Toluene	≤30	≤20
Ethyl Benzene	≤30	≤20

SPCC's	Avg. RF
Chloromethane	≥0.10
1,1-Dichloroethane	≥0.10
Bromoform	≥0.10
1,1,2,2-Tetrachloroethane	≥0.30
Chlorobenzene	≥0.30

Linearity: If the % RSD of any compound is 15% or less, then the relative response factor may be used for quantitation.

When the RSD exceeds 15%, the plotting and/or visual inspection of the calibration data can be a useful diagnostic tool. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.

If a %RSD greater than 30 percent is measured for any CCC, then corrective action to eliminate a system leak and/or column active sites is required before reattempting calibration.

If the %RSD of ANY compound exceeds 15% then one of the following options must be applied to the GC/MS initial calibration in this situation, or a new initial calibration must be performed. The analyst may use the following option to determine the usability of an initial calibration if any compound exceeds the 15% RSD requirement.

1. Narrow the calibration range until the response is linear. If the low standard is below the estimated quantitation limit (i.e., for the poor purgers in a commercially available prepared standard mix), then this standard may be dropped. If a point lower than the reporting limit is not used for the poor purging compounds a 5 point must be added within the same time window at a higher concentration. At no time can any compound have less than 5 points. Recalculate the RSD without this standard to see if the RSD meets the QC limit. It would be recommended that a new standard be prepared at a concentration between the existing fourth and fifth calibration standards, analyzed, and a new RSD calculated with all five points.
2. Use a linear calibration (first order) that does not pass through the origin. This would be achieved by performing a linear regression of the instrument response versus the concentration of the standards. The line should not be forced through the origin and the origin should not be included as a sixth calibration point. In order to be used for quantitative purposes, the correlation coefficient must be greater than or equal to 0.99.

These curves are to be verified by a second source calibration standard known as the initial calibration verification (ICV) standard. This standard shall be prepared near the mid-point of the calibration standard. The second source check must meet all the CCV requirements listed in section 8.4.2.5. If all

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requirements are not met for the second source check, then corrective action would be required; up to and including reanalysis of the initial calibration.

3. If options 1 and 2 fail the initial calibration criteria, adjust the instrument and/or perform instrument maintenance until the RSD of the calibration meets the 15% QC limit. This option would apply in those instances where a linear instrument response is expected. (New calibration is required after maintenance.)

9.4.2.6 Daily GC/MS calibration verification. Prior to the analysis of samples and then every 12 hours, inject 50ng of the 4-bromofluorobenzene standard. The resultant mass spectra for the BFB must meet all of the criteria. The 12-hour clock starts at the injection of the BFB. The last sample must be injected within 12 hours of the BFB injection.

The initial calibration curve should be verified for all method target analytes every 12 hours. This is accomplished by analyzing a continuing calibration verification (CCV) standard at a concentration near the midpoint concentration for the working range of the GC/MS.

System Performance Check Compounds (SPCCs) (this is the same check that is applied during the initial calibration). A system performance check must be performed each 12 hour shift. If the minimum relative response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. After the system performance check is met, the percent drift is calculated for each target analyte.

All project-specific analytes must demonstrate a % drift <20%. If project-specific information is not known, the CCCs must meet this criteria. Under no conditions could any method target analyte exceed 40%. If this criteria is exceeded, corrective action is required. If no source of the problem can be determined after corrective action has been taken, a new five point calibration MUST be generated. This criterion MUST be met before quantitative sample analysis begins.

$$\% \text{ Drift} = \frac{(C_1 - C_0)}{C_1} \times 100$$

Where: C_1 = calibration check compound standard concentration
 C_0 = measured concentration using selected quantitation method.

The internal standard responses and retention times in the check calibration standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last calibration verification, the chromatographic system must be inspected for malfunctions and corrections must be made as required. If the EICP area for any of the internal standards changes by a factor of two (-50% to +100%) from the last daily calibration standard check, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning are necessary.

- 9.4.3 If time remains in the 12 hour time period after meeting the acceptance criteria for the initial calibration, samples may be analyzed. It is not necessary to analyze a continuing calibration

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standard, if the initial calibration meets the calibration acceptance criteria above. A method blank is necessary.

9.4.4 If time does not remain in the 12 hour period beginning with the injection of BFB, a new injection of BFB must be made. If the new injection meets the ion abundance for BFB, then a continuing calibration standard may be injected.

9.4.5 The response factors for the continuing calibration standard must meet the criteria given in paragraph 8.4.2.5 prior to the analysis of any blanks or samples.

9.5 GC/MS Analysis

9.5.1 To determine whether a soil is to be analyzed as a low or medium level is at the discrimination of the analyst. If peaks are saturated or out of calibration range at 200 ug/kg, a smaller size of sample may be analyzed. However, the smallest sample size permitted is 1 g. If smaller than 1 g sample size is needed to prevent saturation, the medium level method must be used.

9.5.2 Low Level Soil Method

The low level soil method is based on purging a heated soil sample mixed with reagent water containing the ISTD/SURR. Refer to SOP 6.05. Analyze all method blanks and standards under the same conditions as the samples.

9.5.2.1 The GC/MS system should be set up under the same conditions as in section 9.4. This should be done prior to the preparation of the sample to avoid the loss of volatiles from the standards to the samples. A heated purge calibration curve must be prepared and used for the quantitation of all samples analyzed with the low-level method. Follow the initial and daily calibration instructions, but increase the purge temperature to 40°C.

9.5.2.2 To prepare the reagent water containing the ISTD/SURR standards, remove the plunger from the 10 ml gas tight syringe and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume of the water to 10 ml. Back off plunger to allow space for IS standard/surrogate. Add 5 ul of the ISTD/SURR standard to the syringe through the valve.

9.5.2.3 The sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh 5 g into a tared purge device to the nearest 0.1 g.

9.5.2.4 (optional) Immediately after weighing the sample, weigh 5-10 g of the soil into a tared drying boat. Determine the percent moisture by drying overnight at 105°C. Allow to cool in a desiccator before weighing. Concentrations of individual analytes will be calculated using the dry weight basis.

$$\% \text{ Moisture} = \frac{(\text{Wet Wt.} - \text{Dry Wt.})}{\text{Wet Wt.}} \times 100\%$$

9.5.2.5 Add the spiked reagent water to the purge device and connect the device to the purge and trap system.

9.5.2.6 Optimize by heating the sample up to 40°C for up to 2 minutes and then purging for up to 11 minutes (may vary per instrument).

9.5.2.7 At the conclusion of the purge time, desorb the sample off the trap and begin the gas chromatographic temperature program. Concurrently, introduce the trapped

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materials to the gas chromatographic column by rapidly heating the trap to 180-200°C (see section 8.1) while backflushing the trap with an inert gas between 35 and 40 ml/min for two minutes.

9.5.2.8 After desorbing the sample off the trap, bake the trap at up to 230°C for up to 10 minutes. When the trap is cooled, the trap is ready for the next sample.

9.5.2.9 To prepare matrix spike and matrix spike duplicate for low level soils, add 5 ul of the matrix spike solution to the 10 ml of water added to each of the two aliquots of the soil from the sample chosen for the spiking. The concentration for a 5 g sample would be equivalent to 100 ug/Kg of each matrix spike compound.

9.5.2.10 A volatile blank must be analyzed at least once every 20 samples or at the beginning of each work day, whichever is more frequent.

- For low level soil samples, a volatile method blank consists of 5g of purified sand.
- Solid matrix added to reagent water, spiked with the ISTD/SURR and carried through the analytical procedure.
- An acceptable volatile blank for low level soil samples must contain analytes less than the reporting limit. The sample value is not to be corrected for the blank value. All samples analyzed using Army Corp. CDQMP must be N.D. less than 1/2 the reporting limit. All samples analyzed using the Navy Guidance document must be clean to the MDL.
- All volatile analysis associated with a blank that does not meet the blank requirements above must be reanalyzed.

9.5.3 Medium Level Soil Method

The medium level soil method is based on extracting the soil with methanol. An aliquot of the methanol extract is added to reagent water containing the ISTD/SURR standards. The reagent water containing the methanol extract is purged at ambient temperature. All samples where 5g sample was analyzed and the target compounds were out of calibration range must be analyzed using the medium level method.

9.5.3.1 The GC/MS system should be set up as in section 9.4. This should be done prior to the addition of the methanol extract to reagent water. Because the methanol extract and reagent water mixture is purged at ambient temperature, the instrument performance check, initial calibration, and continuing calibration for water samples may be used for analysis of medium soil sample extracts.

9.5.3.2 The sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh 4g into a tared 40 ml VOA to the nearest 0.1 g. Add surrogate standards at this point then 10 mL of methanol to the VOA.

9.5.3.3 If the sample was submitted as a medium level, start with 50 ul of the extract into 10 ml of reagent water in a 10 ml syringe spiked with the ISTD solution. If a greater dilution is required, lesser amounts of the methanol extract is acceptable.

9.5.3.4 Attach the syringe to the syringe valve on the purging device. Open the syringe valve and inject the water/methanol sample to the purging chamber.

9.5.3.5 Proceed with the analysis as outlined in sections 9.5.2.6 - 9.5.2.9. Analyze all method blanks on the same instrument as the samples.

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- 9.5.3.6 For precision, duplicate samples must be prepared.
(optional – MS/MSD) To prepare a matrix spike and matrix spike duplicate for the medium level soil samples, add 9.9 ml of methanol, .01 ml of the matrix spiking solution to each of the two aliquots of the soil sample chosen for spiking. This results in a 1000 ug/Kg concentration of each matrix spike compound when added to a 5 g sample. Add 10 ul of the methanol extract to 5 ml of reagent water for purging as described in paragraph 9.5.3.5.

9.5.4 Water Samples

- 9.5.4.1 All water samples must achieve equilibrium with the ambient temperature before analysis.
- 9.5.4.2 Each GC/MS system must be calibrated with a 5 point calibration curve using 10mL volume.
- 9.5.4.3 Remove the plunger from a 10mL syringe. Carefully pour the sample into the syringe barrel to just short of over-flowing. Replace the plunger and vent all air while bringing to a final volume of 10mL. Check the pH.
- 9.5.4.4 If a dilution is necessary, the dilution will be made prior to the analysis using DI water. The dilution factor will be calculated using the following equation.

$$DF = \frac{SV}{(SV + DI)}$$

Where: DF = Dilution Factor
SV = Sample Volume (mL)
DI = DI Volume (mL)

- 9.5.4.5 Add 5uL of the internal standard/surrogate solution through the valve bore of the syringe. After addition, the spike concentration of internal standards will be 25 ug/L and 50ug/L for surrogates.
- 9.5.4.6 Attach the syringe to the valve on the auto sampler and introduce the sample.
- 9.5.4.7 Purge sample at ambient temperature.
- 9.5.4.8 After purging, the sample/QC/standard the trap is back flushed with Helium while rapidly heating the trap up to 180-200°C.
- 9.5.4.9 After the sample has been introduced into the GC, the trap is baked up to 10 minutes at up to 230°C, depending on the instrument. The needle is wiped with methanol. Samples with high level of contamination may require additional instrument blanks analyzed to clean the sparge position used.
- 9.5.4.10 To prepare a laboratory control spike add 5ul of Matrix Spike and IS/Surrogate solution to 10 mL of DI water giving a spike concentration of 50ug/L.
- 9.5.4.11 To prepare the matrix spike add 5ul of the matrix spike solution to 10 mL of the sample chosen to spike. The spike concentration is 50ug/L.
- 9.5.4.12 A method blank must be analyzed at least once every 12 hours tune. **USACE requires that method blanks be run per loading of the purge and trap unit or every 4 hours to monitor room conditions.**
- For water samples, a volatile method blank consists of a 10 mL volume. Spiked with surrogate/internal standard.

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- All method blanks must be free of target analytes at or below 2X MDL. Refer to section 10. All samples associated with a blank exceeding QA/QC limits must be reanalyzed.

9.5.5 Air Analysis

- 9.5.5.1 Allow air sample to achieve equilibrium with ambient air temperature.
- 9.5.5.2 Air samples are analyzed using water methodology (see section 9.5.4)
- 9.5.5.3 The sample is introduced by adding 10 mL of DI water with 5uL of the internal standard/surrogate mix to the sparge vessel. Using a 10-mL gas tight syringe, transfer 10 mL of sample into the purge vessel.
- 9.5.5.4 Close the valve and purge the sample for 10 minutes using same parameters as water sample (see section 9.5.4)
- 9.5.5.5 If a sample requires dilution, use less aliquot of air. The minimum volume of air that can be used is 1mL.

9.6 Qualitative Analysis

- 9.6.1 The target compound's shall be identified by an analyst competent in the interpretation of mass spectra by comparison of the sample spectrum to the mass spectrum of a standard of the suspected compound. Two criteria must be satisfied to verify the identifications:
 - 1. elution of the sample component at the relative retention time as the standard component
 - 2. correspondence of the sample component and standard component mass spectra
- 9.6.1.1 For establishing correspondence of the GC relative retention time (RRT), the sample component must compare within 0.06 RRT units of the RRT of the standard component. For reference, the standard must be run in the same 12-hour time period as the sample. If coelution of interference components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles (EICP) for the ions unique to the component of interest.
- 9.6.1.2 For comparison of standard and sample component mass spectra, mass spectra must be obtained on the GC/MS system. Once obtained, these standard spectra may be used for identification purposes, only if the GC/MS meets the daily instrument performance requirements for BFB. These standard spectra may be obtained from the run used to obtain reference RRT's.
- 9.6.1.3 The requirements for qualitative verification by comparison of mass spectra after background correction with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of the method. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity or any one over 30% relative intensity if less than three such ions occur in the ref. spectrum. Compounds should be identified as percent when criteria below is met.
 - All ions present in the standard mass spectra at a relative intensity greater than 10% must be present in the sample spectrum.
 - The relative intensities of ions specified in paragraph 9.3.6 must agree within $\pm 20\%$ between the standard and the sample spectra. (Example: For an ion with

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an abundance of 50% in the standard the corresponding sample ion abundance must be between 30 and 70%).

- Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. All compounds meeting the identification criteria must be reported.

9.6.1.4 If a compound cannot be verified by all of the criteria in paragraph 9.6.1.3, but in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then report that identification and proceed with quantification in paragraph 9.7.

9.6.2 A library search shall be executed for non-target sample components for the purpose of tentative identified compounds (TIC's), the most recent release of the NIST/EPA/MSDC mass spectral library, containing 75,000 spectra, shall be used. Computer generated library search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

9.6.2.1 Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. Computer generated library search routines must not be normalized that would misrepresent the library of unknown spectra when compared to each other.

9.6.2.2 Guidelines for making TIC's.

- Relative intensities of major ions in the reference spectrum should be present in the sample spectrum.
- The relative intensities of the major ions should agree within $\pm 20\%$.
- Molecular ion present in reference spectrum should be present in sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.
- Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination of co-eluting compounds. Data system library reduction programs can sometimes create these discrepancies.

9.6.2.3 If in the technical judgment of the mass spectral interpretation specialist, no valid TIC's can be made, the compound should be reported as unknown. The mass spectral specialist should give additional classification of the unknown compound, if possible (i.e., unknown alkaline, unknown aromatic, unknown acid type, unknown chlorinated compound). All compounds reported as TIC's will be flagged as estimate.

9.7 Quantitative Analysis

9.7.1 Target components identified shall be quantified by the internal standard method. The internal standard used shall be that which is assigned. The EICP of the characteristic ions of analytes are used.

1,4-Difluorobenzene	1,4-Dichlorobenzene-d4	Pentafluorobenzene	Chlorobenzene-d5
Carbon Tetrachloride	1,2,3-Trichlorononane	Vinyl Chloride	1,3-Dichlorononane

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1,2-Dibromoethane	p-Isopropyltoluene	Bromoethane	Tetrachloroethene
1,1,2-Trichloroethane	Propylbenzene	1,1-Dichloroethene	2-Hexanone
Trans-1,3-Dichloropropene	1,1,2,2-Tetrachloroethane	Acetone	Chlorobenzene
Toluene	2-Chlorotoluene	Methylene Chloride	Ethylbenzene
Toluene d8 (surr)	Tert-Butylbenzene	Trans-1,2-dichloroethene	Xylene
1,2-Dichloroethane	1,2,4-Trimethylbenzene	2-Butanone	Dibromochloromethane
1,2-Dichloroethane d4(surr)	sec-Butylbenzene	cis-1,2-Dichloroethene	1,1,1,2-Tetrachloroethane
Dibromomethane	1,2-Dichlorobenzene	Bromochloromethane	Styrene
Bromodichloromethane	1,3-Dichlorobenzene	Dichlorodifluoromethane	Bromoforn
2-Chloroethylvinylether	1,4-Dichlorobenzene	Chloromethane	
cis-1,3-Dichloropropene	Butylbenzene	Chloroethane	
4-methyl-2-pentanone	Naphthalene	Vinyl acetate	
1,1,1-Trichloroethane	1,2-Dibromo-3-chloropropene	Chloroform	
1,1-Dichloropropane	1,2,4-Trimethylbenzene	2,2-Dichloropropane	
Trichloroethene	1,3,5-Trimethylbenzene	Carbon Disulfide	
Benzene	1,2,3-Trichlorobenzene	Trichlorotrifluoroethane	
1,2-Dichloropropane	4-Chlorotoluene		
	Hexachlorobutadiene		
	Isopropylbenzene		
	Bromofluorobenzene (surr)		
	Bromobenzene		

9.7.2 Internal standard responses and retention times in all standards must be evaluated during or immediately after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last daily calibration standard, the gas chromatograph must be inspected for malfunctions, and corrections made as required. The EICP of the internal standard must be monitored and evaluated for each sample, blank, matrix spike, and matrix spike duplicate. If the EICP area for any internal standard changes by more than a factor of two (-50% to +200%), the mass spectrometer system must be inspected for malfunction and corrections made as appropriate. When corrections have been made, reanalysis of samples analyzed while the system was malfunctioning is necessary.

9.7.2.1 If after reanalysis, the EICP areas for all the internal standards are inside the limits (-50% to +200%), then the problem of the first analysis is considered to have been within the control of the laboratory. Therefore, submit only the data from the analysis with the EICP's within the limits. This is considered the initial analysis.

9.7.2.2 If the reanalysis of the sample does not solve the problem, i.e. the EICP areas are outside the limits for both analysis, then both analysis are considered valid. Distinguish between the initial analysis and the reanalysis, using all quantitation from the first analysis. The data must be flagged with the following footnote:
"Internal standard was outside of QA/QC limits due to matrix interference. Results bias high".

9.7.3 The average response factor from the ICAL is used to calculate the concentration in the sample. Use the RF as in paragraph 9.4.2.3 and the equations below.

Water concentration ug/L

$$C = \frac{(A_x)(I_s)(DF)}{(A_{is})(RF)(V_1)}$$

Where: C = Concentration (ug/L)
 V₁ = Volume of water purged in mL.
 A_x = Area of the characteristic ion for the compound to be measured.
 A_{is} = Area of the characteristic ion for the specific internal standard.
 I_s = Amount of internal standard added in nanograms.
 DF = Dilution Factor

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RF = Average Response Factor

Low Soils

$$C = \frac{(A_x)(I_s)}{(A_{is})(RF)(W)}$$

Where: C = Concentration (ug/Kg)

 A_x = Area of the characteristic ion for the compound to be measured. A_{is} = Area of the characteristic ion for the specific internal standard. I_s = Amount of internal standard added in nanograms.

W = Weight of sample added to purge tube, in grams.

RF = Average Response Factor

$$D \text{ (Dry Weight)} = \frac{100 - \% \text{ Moisture}}{100}$$

Medium Soil

$$C = \frac{(A_x)(I_s)(V_t)(1000)}{(A_{is})(RF)(V_i)(W)}$$

Where: A_x = Area of the characteristic ion for the compound to be measured. A_{is} = Area of the characteristic ion for the specific internal standard. I_s = Amount of internal standard added in nanograms.

RF = Average Response Factor

 V_t = Total volume of the methanol extract in milliliters. (Note: The volume is 10 milliliters.) V_i = Volume of aliquot of the methanol extract in microliters added to the reagent water for purging.

W = Weight of soil extracted, in grams. Note: The weight should be 4 grams.

1000 = The factor of 1000 in the numerator converts the value of V from ml to ul.

- 9.7.4 An estimated concentration for non-target components tentatively identified shall be determined by the internal standard method. For quantification, the nearest internal standard free of interference's shall be used.

The formula for calculating concentrations is the same as in paragraph 9.7.3. Total area counts from the total ion chromatograms are to be used for both the compound of to be measured and the internal standard. An Avg. RF of one (1) is to be assumed. The resulting concentration shall be labeled an estimated concentration. This calculation shall be done on all TIC's including the ones that are "unknown".

- 9.7.5 Xylenes (o-, m-, and p- isomers) are to be reported as Xylenes, total. The concentration of all Xylenes isomers must be added together to give the total.

- 9.7.6 Both trans and cis stereoisomers of 1,2 Dichloroethene are to be reported as separate analytes unless requested otherwise by client.

10.0 QUALITY ASSURANCE

- 10.1 If the on-column concentration of any compound in any sample exceeds the initial calibration range, the sample must be diluted, and the sample repurged. Guidance in performing dilutions, and exceptions to the requirement are given below.

- 10.1.1 Use the result of the original analysis to determine the approximate dilution factor required to get the largest analyte peak within the upper half of the initial calibration range.

- 10.1.2 For the Xylenes and the 1,2-Dichloroethene isomers are quantified as separate peaks, the calibration of each peak should be considered separately. i.e. a diluted analysis is not

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required for the total Xylenes unless the concentration of either peak separately exceeds 100 ug/L (200 for m & p).

- 10.2 Calculate the recovery of each system monitoring compound in all samples, blanks, matrix spikes, and matrix spike duplicates. Determine if the recovery is within the surrogates recovery limits.

- 10.2.1 Calculate the concentrations of the surrogates using the same equations as used for target compounds. Calculate the recovery of each surrogate as follows:

$$\% R = \frac{C F}{C S} \times 100$$

Where: %R = Percent Recovery
 CF = Concentration Found
 CS = Concentration Spiked

- 10.3 A matrix spike and matrix spike duplicate must be performed for each group of samples of a similar matrix, for the following, whichever is most frequent:

- Each 20 field samples
- Each matrix of samples
- Every working day

- 10.3.1 Calculate the concentrations of the matrix spike compounds using the same equations as used for target compounds. Calculate the recovery of each matrix spike compound as follows:

$$\% R = \frac{C F}{C S} \times 100$$

Where: %R = Percent Recovery
 CF = Concentration Found
 CS = Concentration Spiked

- 10.3.2 Calculate the relative percent difference (RPD) of the matrix spike and matrix spike duplicate as follows:

$$RPD = \frac{ABS(MSR - MSDR)}{AVG(MSR + MSDR)} \times 100$$

Where: MSR = Matrix Spike Recovery
 MSDR = Matrix Spike Duplicate Recovery

- 10.3.3 The limits of the matrix spike compound are only advisory, no further action is required, however, frequent failures to meet the requirements must be investigated.

- 10.4 Determine the concentrations of any target compounds detected in the volatile method blank, using the equations in paragraph 9.7.3.

11.0 CORRECTIVE ACTION

This section encompasses corrective action procedures initiated by failure of quality control mechanisms during a batch run, such as blanks, LCS's, and matrix spike/duplicate recoveries out of limits. Each analytical method may have additional control samples. Corrective actions for failure of these controls is also be included.

NOTE: Corrective actions on QC failures may vary to meet specific client or project requirements.

- 11.1 General Corrective Action Procedures

- 11.1.1 Method Blank Samples.

- 11.1.1.1 Acceptance criteria is $ND \leq 2X MDL$.

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11.1.1.2 Corrective Action.

1. Check calculations.
2. Re-analyze the blank.
3. If the concentration of a targeted analyte in the method blank is less than 2X MDL, then a corrective action form must be submitted and associated data may be reported without qualification. Note that the re-analyzed blank must meet the time and analytical constraints to be in the same analytical batch as the failed blank.
4. If the concentration of a targeted analyte in the method blank is greater than 2X the MDL but less than the client's reporting limit, corrective action is required, but the associated data may be reported without qualification.
5. If the concentration of a targeted analyte in the method blank is greater than 2X MDL and greater than the client's reporting limit, and the associated sample values are more than 10 X the concentration present in the blank, corrective action is required, and the associated data must be flagged.
6. If the concentration of a targeted analyte in the method blank is greater than 2X MDL and greater than the client's reporting limit, and the associated sample values are less than 10 X the concentration present in the blank, corrective action is required, and the sample batch must be re-processed.

11.1.2 Matrix Spike.

11.1.2.1 Acceptance Criteria - For accuracy and precision, refer to QA Manual, individual SOPs or project requirements.

11.1.2.2 Corrective Action.

1. Check calculations.
2. Assess data to determine whether spike results are visually attributable to a matrix effect or are the result of other problems in the analytical process.
3. If the recoveries are less than the acceptable limits, then reanalyze once and a corrective action is required. If the re-analysis fails, then corrective action is required, the associated data must be flagged, but re-preparation and re-analysis is not required. See Section 11.1.4 (LCS).

11.1.3 Matrix Spike Duplicate.

11.1.3.1 Acceptance Criteria - For accuracy and precision, refer to QA Manual, individual SOPs or project requirements.

11.1.3.2 Corrective Action.

1. Check calculations.
2. Assess data to determine whether spike results are visually attributable to a matrix effect or are the result of other problems in the analytical process.

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3. If the recoveries are less than the acceptable limits, then reanalyze once and a corrective action is required. If the re-analysis fails, then corrective action is required, the associated data must be flag, but re-preparation and re-analysis is not required. See Section 11.1.4 (LCS).

11.1.4 Laboratory Control Samples (LCS).

11.1.4.1 Acceptance Criteria - For accuracy and precision, refer to QA Manual, individual SOPs or project requirements.

11.1.4.2 Corrective Action.

1. Check calculations.
2. Re-analyze the LCS. If the re-analysis passes acceptance criteria, then a corrective action is required but the data can be reported without qualification.
3. If the re-analysis fails acceptance criteria, then corrective action is required and the sample batch must be re-processed (including preparation and analysis).
4. Note that precision can be obtained either from the LCS/LCSD or the MS/MSD with the following caveat: If an LCSD is run, then precision must come from the LCS/LCSD, otherwise precision is taken from the MS/MSD.

11.1.4.3 Agency Specific Requirements

1. For all USACE projects, the LCS (and LCSD if applicable) shall be spiked with all target analytes of interest (by project).
2. In lieu of the issuance of default limits from USACE, the laboratory will
 - a) Establish its own default limits if not specified by the reference method.
 - b) Establish statistical limits when enough data points become available for each compound.

11.2 Additional Requirements Specific to Volatile Organics by GC/MS.

11.2.1 System Blank

11.2.1.1 Acceptance Criteria - $\leq 2X$ MDL.

11.2.1.2 Corrective Action.

1. Run until system is clean.

11.2.2 Surrogate Spike.

11.2.2.1 Acceptance Criteria - refer to QA Manual, individual SOPs, or project requirements. If the recovery of any one of the surrogates is not within the limits in Table #2, refer to 11.2.2.2.

Table #2

Surrogate	Water/Medium Level	Soil	Reference
1,2-Dichloroethane-d4	76-114	70-121	1
Toluene-d8	88-110	81-117	1
4-Bromofluorobenzene	86-115	74-121	1
Dibromofluoromethane	76-114	70-121	1

11.2.2.2 Corrective Action.

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1. Evaluate the QC samples. If the appropriate QC samples pass acceptance criteria, then continue to step 2.
2. Check surrogate calculations and surrogate peak integration.
3. Evaluate data with respect to other samples in the batch. If all samples have the same type of surrogate failure, then the surrogate solution is possibly suspect or there is some matrix interference. If necessary, re-analyze the sample one time. If the surrogate meets acceptance criteria, then a corrective action is required and the sample can be reported without qualification.
4. For purgeables, if the surrogate fails the re-analysis, then matrix effects can be assumed. A corrective action is required and the data must be flagged. If the re-analysis passes then a corrective action is required and the sample can be reported without qualification.

11.2.3 Internal Standard.

11.2.3.1 Acceptance Criteria - RT must be within 30 sec. of CCV; IS area in the sample must be within a factor of 2 of the IS in the CCV.

11.2.3.2 Corrective Action.

1. Check instrument sensitivity.
2. Evaluate data.
3. Reanalyze sample or standard once.
4. Narrate any outliers.

11.2.4 Instrument Quality control

11.2.4.1 Performance Check Standard (BFB)

All ion abundance criteria must be met before any analysis can proceed. All Samples, QC, Standards, and conformation samples must be within the 12 hour tune window. If QC is outside of the window, the whole batch fails.

11.2.4.2 The ICV must be within control limits for analysis to proceed.

11.2.4.3 All CCV must be within QA/QC guidelines. If a CCV is outside of QA/QC limits all samples, blanks, and QC must be reanalyzed.

11.2.4.4 Response factors for SPCCs must be evaluated and pass for all CCV. If any SPCC does not meet the minimum response, the system must be taken down and the problem corrected. The analyst should evaluate the integrity of the standard as well as inlet contaminants.

11.2.4.5 If the criterion is not met for any on CCC, corrective action must be taken. If the problem can't be corrected, a new 5-point must be analyzed.

12.0 DATA REVIEW

- 12.1 All data, QC, pertinent information (i.e., corrective actions / non-conformances), and final reports will be compiled into a folder.
- 12.2 A data review checklist will be enclosed in the folder and signed by the chemist.
- 12.3 A second analyst will review the data, initial and date the first page of each chromatogram or analytical report, and sign the data review checklist acknowledging that the information is correctly reported and that all discrepancies have been addressed and corrected.

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12.4 The final report will include signatures of the analyst and reviewer.

13.0 ATTACHMENTS

- I. Run Log & Instructions for filling out Attachment I
- II. Corrective Action Table

AT/JT

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Eff. Date: 08/11/00

Approved By: ***Eric Tam***

Ref : SOP 6.06 & 6.06A

Prep batch # 1

[illegible]

Reviewed By: 18 Date: / /

Page #

FORMS\RNLGCMSSVOAHP#2-1999.DOC

Instructions for filling out Attachment 1

1. Batch #
2. Fill in date
3. Fill in time
4. Fill in file ID
5. Fill in submission number
6. Fill in LIMS ID
7. Fill in client ID
8. Fill in sparger number
9. Fill in matrix
10. Fill in weight/volume
11. Fill in dilution factor
12. Fill in pH
13. Check if run is acceptable
14. Analyst initial
15. Fill in standard ID
16. Fill in comments
17. Line out unused areas
18. Reviewer initial & date

Attachment II

8260B-GC/MS VOLATILES

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Calibration	Frequency	Acceptance Criteria	Corrective Action
Check tune using BFB.	Every 12 hours.	Refer to SOP.	1. Retune. 2. RA BFB.
ICAL-minimum 5-pt (6-pt quadratic). Low std @ RL	Initial/as required	CCCs $\leq 30\%$ RSD SPCCs-Avg. RF ≥ 0.30 (CHBr ₃ -0.25)	1. Evaluate. 2. Recalibrate.
CCV	Every 12 hours or per batch, whichever is less.	CCCs $\leq 25\%$ Difference SPCCs-Avg. RF ≥ 0.30 (CHBr ₃ -0.25)	1. Evaluate. 2. RA. 3. If passes, proceed/report. 4. If fails, recalibrate. 5. RA-affected samples-samples must be bracketed by passing CCVs
QC Sample	Frequency	Acceptance Criteria	Corrective Action
Method Blank	1/batch, 20 samples or less	<RL or refer to QAPP	1. Check calculations. 2. RA. If passes, report. 3. No action if samples ND or $\geq 10x$ MDL. 4. Samples $<10x$, RX/RA.
System blank	As required	<RL	Run until system is clean.
LCS	1/batch, 20 samples or less	Refer to Acceptance Criteria Table.	1. Check calculations. 2. RA. If passes, report. 3. If fails, RX/RA affected samples. 4. Document.
LCSD	1/batch, 20 samples or less	Refer to Acceptance Criteria Table.	1. Check calculations. 2. RA. If passes, report. 3. RA. If passes, report. 4. If fails, RE/RA affected samples, or evaluate MS/MSD for accuracy & precision. 5. Document.
MS	1/batch, 20 samples or less	Refer to Acceptance Criteria Table. or refer to QAPP	1. Check calculations. 2. Evaluate-trends, interferences, 4x rule. 3. If item 2 is not a cause, RX/RA MS/MS 4. Document.
MSD	1/batch, 20 samples or less	Refer to Acceptance Criteria Table. or refer to QAPP	1. Check calculations. 2. Evaluate-trends, interferences, 4x rule. 3. If item 2 is not a cause, RX/RA MS/MSD or 4. Evaluate LCS/LCSD for accuracy/precision. 5. Document.
Surrogate Spike	Every sample	Refer to Acceptance Criteria Table.	1. Check calculations. 2. Evaluate-trends, interferences 3. Reanalyze once. 4. If item 2 is not a cause, RE/RA if one surrogate /fraction exceeds limits. 5. Document.
Internal Standard	Every CCV and sample	RT ≤ 30 sec of IS in CCV. $0.5x < \text{area} < 2x$ of CCV	1. Check instrument sensitivity. 2. Evaluate data, trends-interferences. 3. RA sample. 4. Document.

APPENDIX B

**COPY OF
CHAIN-OF-CUSTODY RECORD**



**2855 Mitchell Drive, Suite 111
Walnut Creek, California 94598
(925) 256-8898 – (925) 256-8998 (fax)**

Chain-Of-Custody

Project Name and Number: _____

Laboratory Name: _____

Date: _____

Project Manager: _____

Address: _____ Contact Name: _____

Page: _____ of _____

Site Location: _____

Phone: _____

[illegible]

Sampled By: _____

Signature: _____

Special Instructions: _____

Send Results to: _____
(w/fax #)

Turnaround Time: _____

Sampler:

Courier/Airbill No.: _____

Relinquished By/Affiliation:

Date: Time:

Received By/Affiliation:

Date: Time:

Original – Laboratory Yellow – Field/Office